Quarterly Progress Report No. 4

Development of Nondestructive Tests for the Evaluation of Bonded Materials

by

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1969 October 20 - 1970 January 19

AVATD-0009-70-CR

Contract No. N00156-69-C-0913

Sponsored by

Advanced Research Project Agency
ARPA Order No. 1247 Amend. 1
Program Code No. 9D10

Submitted by

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I. INTRODUCTION

This report covers the work performed and the results obtained under the subject contract during the quarter 1969 October 20 through 1970 January 19. Data obtained in the surface condition study are presented. Electric field reflectometry, at 1 KHz and 9.8 GHz, and gas-phase ultrasonic transmission methods are detailed as possible methods for nondestructively evaluating substrate surfaces.

II. PROGRESS ACCOMPLISHED

A. Surface Condition Study

All experimental laboratory preparations and evaluations have been completed up to the point of data correlation and analysis. The information has been grouped for each specimen type; butt tensile, core shear, and lap shear. One representative surface, code 3A, for each of 13 different surface conditions within each group, was thoroughly characterized both in the as prepared condition and immediately following sulfuric acid sodium dichromate etch just prior to adhesive bonding. All substrates were measured for bond-line thicknesses. Nondestructive test and destructive test data for the bonded specimens are summarized.

1. Surface Finishes

A total of 390 substrate surfaces were prepared as indicated in Table I. This was sufficient to provide five specimens at two substrates per specimen for each surface condition and type of specimen. Of these, three specimens per surface condition were bonded and tested.

Photomicrographic, surface roughness, and contact angle data are presented on the following pages in grouped fashion for the most beneficial comparisons.

The photomicrographs were taken with a Burke and James "Rembrandt" model camera using an Agfa solinar lens (focal length = 5 cm., f = 3.5). The film used was Polaroid type 52, 4" X 5".

A Taylor-Hobson Tallysurf Recording Profilometer, Model 3, provided the Center-line-average (CLA) and strip chart profile data. Interpretation of the strip charts is aided by Table II.

Contact angle measurements were performed on the Langmuir-style device pictured in Figure 1. The surface to be measured was brought into the plane of the table. A few small drops of distilled water were placed on the surface to form one highly-domed drop. While sighting along the juncture of the droplet and the surface via a surface mirror attached to the protractor, the protractor was adjusted until the axis of the arm was aligned exactly with the three-phase point. This operation was found to be most important to accuracy and reproducibility. The observed light-extinction angle was read from the protractor.

TABLE I

SURFACE ROUGHNESS PREPARATION

NOMINAL ROUGHNESS	BUTT TENSILE	CORE SHEAR	LAP SHEAR
1 RMS	Paper Lapped	Paper Lapped	Paper Lapped
5 RMS	Paper Lapped	Paper Lapped	Paper Lapped
7 RMS	Paper Lapped	Paper Lapped	Paper Lapped
10 RMS	Paper Lapped	Paper Lapped	Paper Lapped
20 RMS	Turned	Paper Sanded	Paper Sanded
40 RMS	Paper Sanded	Paper Sanded	Turned
80 RMS	Turned	Paper Sanded	Turned
110 RMS	Turned	Turned	Turned
150 RMS	Turned	Turned	Turned
Fine Grit Low Pressure	140 Mesh Silica 20 psi	140 Mesh Silica 20 psi	140 Mesh Silica 20 psi
Fine Grit High Pressure	140 Mesh Silica 80 psi	140 Mesh Silica 80 psi	140 Mesh Silica 80 psi
Coarse Grit Low Pressure	80-100 Mesh Al_2O_3 20 psi	80-100 Mesh Al_2O_3 20 psi	80-100 Mesh Al_2O_3 20 psi
Coarse Grit High Pressure	80-100 Mesh Al_2O_3 80 psi	80-100 Mesh Al_2O_3 80 psi	80-100 Mesh Al_2O_3 80 psi

TABLE II

STANDARD RANGE AND MAGNIFICATION FACTORS

Switch Position	Magnification	Full Scale Chart 2 ins. or 5 cms.		Each Small Division Represents		Full Scale C.L.A. Index	
		Micro-inches	Microns	Micro-inches	Microns	Micro-inches	Microns
1	1,000	2,000	50	100	2.5	200	5.0
2	2,000	1,000	25	50	1.25	100	2.5
3	5,000	400	10	20	0.5	40	1.0
4	10,000	200	5	10	0.25	20	0.5
5	20,000	100	2.5	5	0.125	10	0.25
6	50,000	40	1	2	0.05	4	0.1

1 Micro-inch =.000.001 in. 1 Micron = 0.001 mm. = 40 micro-inches

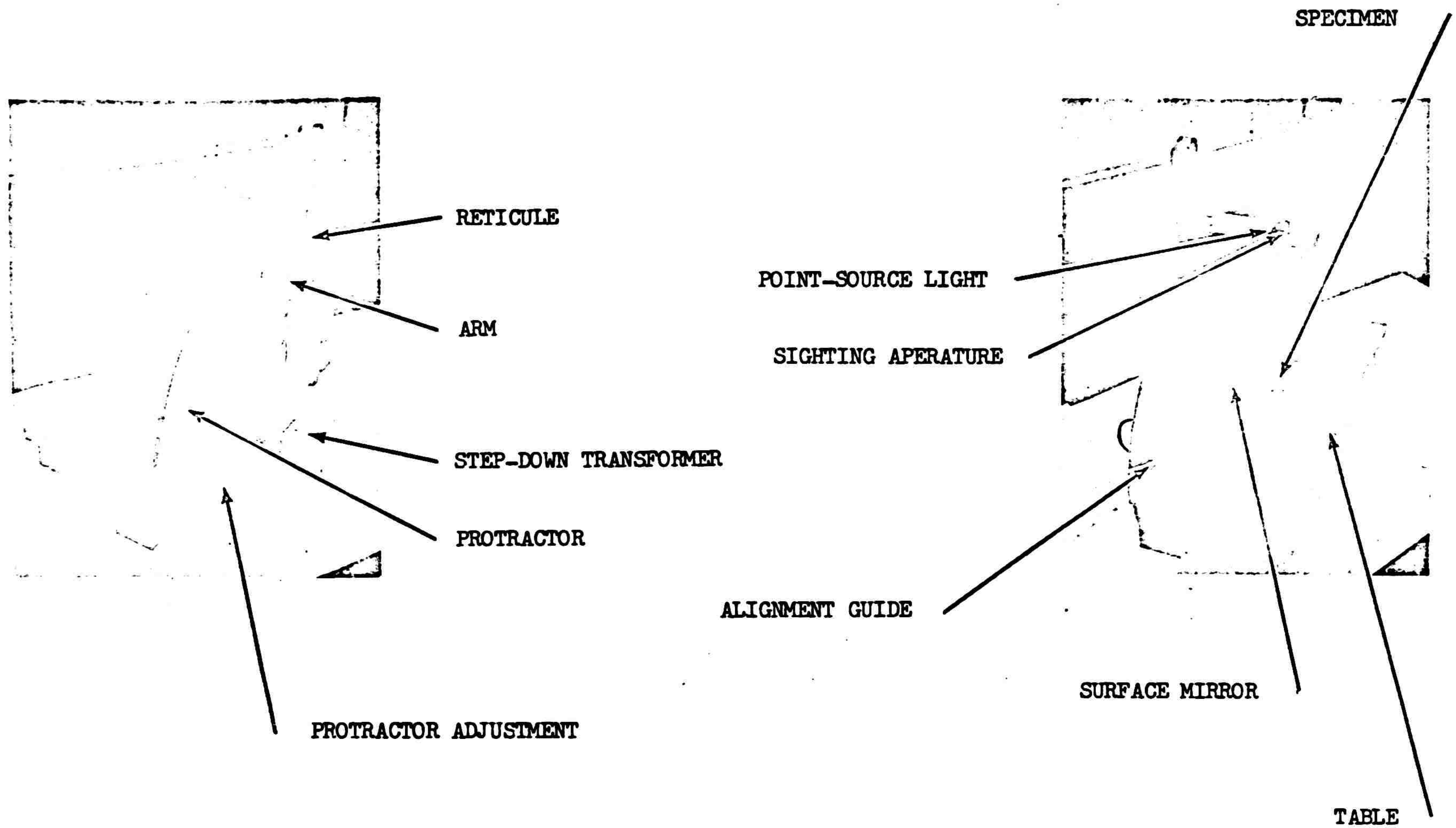


FIGURE 1. CONTACT-ANGLE MEASURING DEVICE DEVELOPED AT AVCO FOR SURFACE CHARACTERIZATION SPECIMENS (AFTER LANGMUIR)

For highly polished, pure-element surfaces, a single contact angle is reported in the literature. In less-perfect cases advancing and receding contact angles are reported. Investigation of the hydrodynamic and gravitational forces involved in the cited measurements, as they relate to the distinctly rough surfaces being studied under this contract, revealed that many important forces act to modify the basic surface-free-energy attractions. Our own early observations revealed other parameters which affect the light extinction point determination, such as droplet shape and "lay" of the surface texture.

A satisfactory technique for performing the contact angle measurements, in consideration of both the theoretical and practical aspects, was developed. By adding water to an existing droplet in small increments the droplet was brought to its maximum contact angle, beyond which, the droplet would "jump" outward to a lower contact angle. Conversely, by removing small increments of water from an existing droplet, a minimum contact angle could be obtained, just prior to the droplet retracting in a "jump" motion. In all cases the test surfaces were horizontal. The results were found to be reproducible and properly related to surface energetics. The data is reported in a group of three values, θ maximum, θ minimum, and the angular difference between them, $\Delta\theta$.

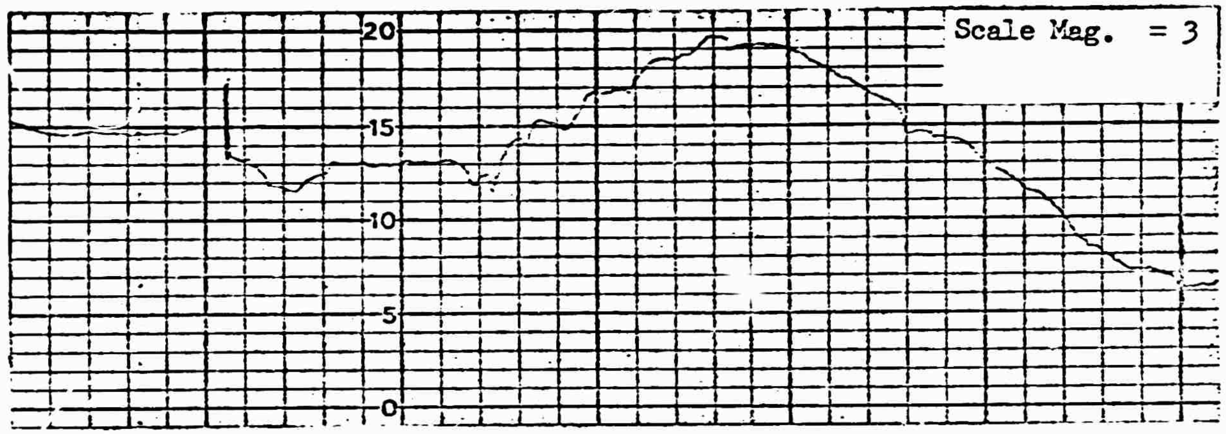
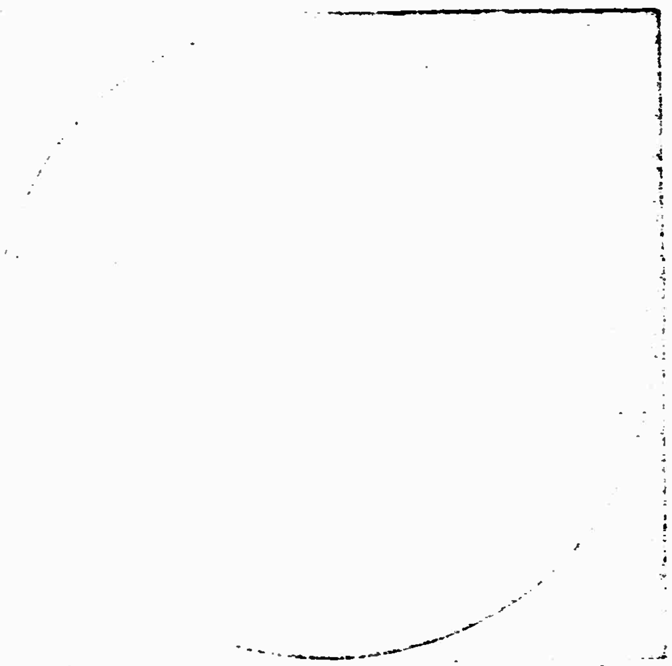


Photo Mag. = 4.4X

CLA = 1.2
RMS = 1.3

1 BT-3A (M) Before Etch

θ MAX	71.5
θ MIN	21.5
$\Delta\theta$	50.0

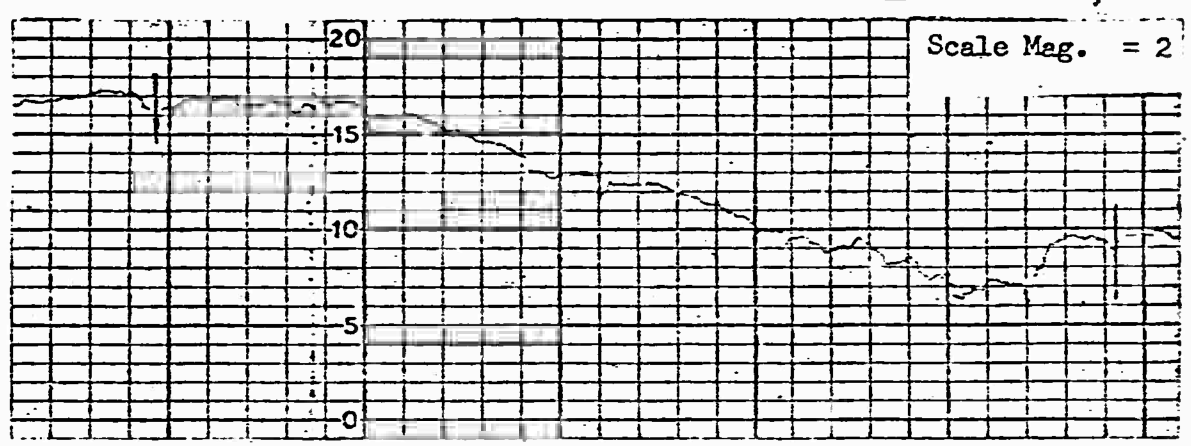


1 BT-3A (E) After Etch

Photo Mag. = 4.5X

CLA = 10.0
RMS = 11.1

θ MAX	45.5
θ MIN	9.0
$\Delta\theta$	36.5



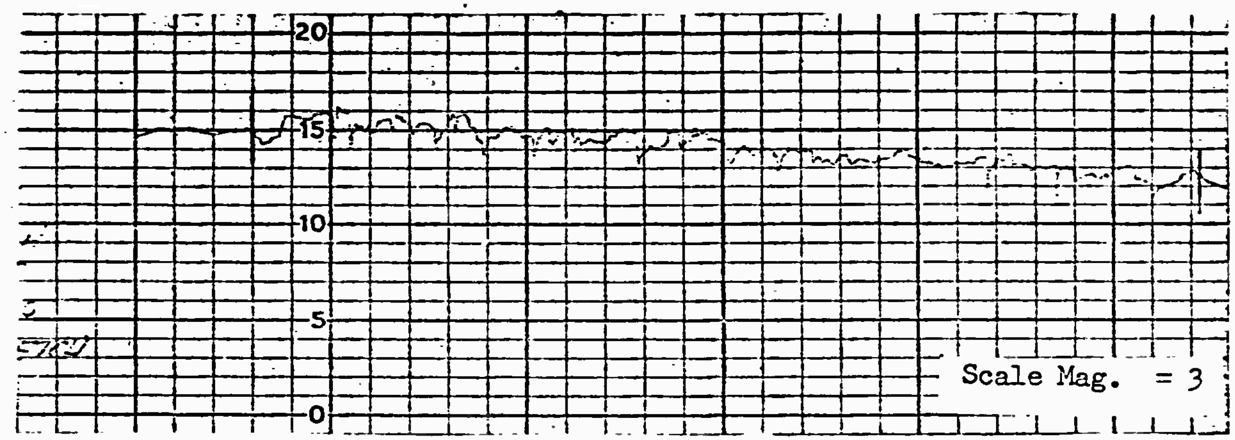


Photo Mag. = 4.4X

CLA = 6.0
RMS = 6.6

5BT-3A (M) Before Etch

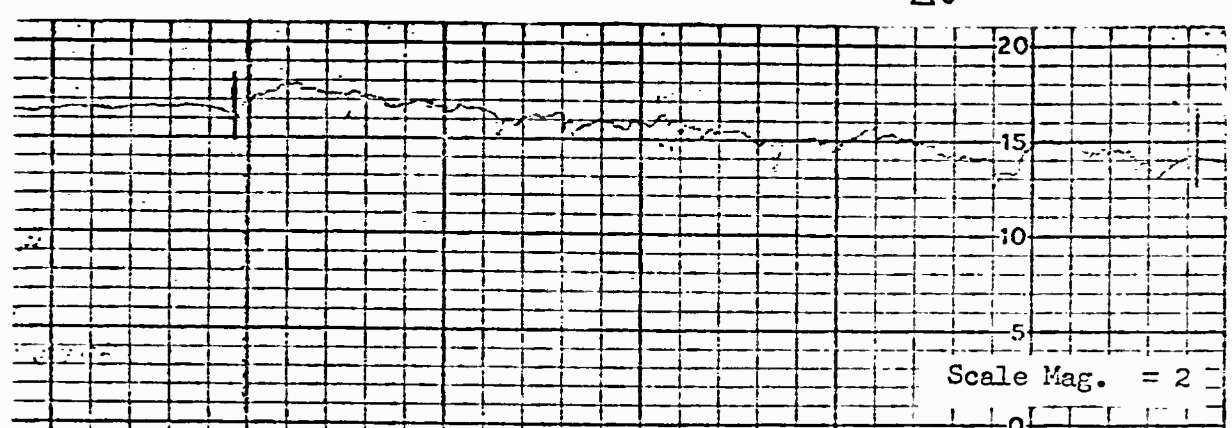
θ MAX = 79.5
θ MIN = 25.0
$\Delta\theta$ 54.5

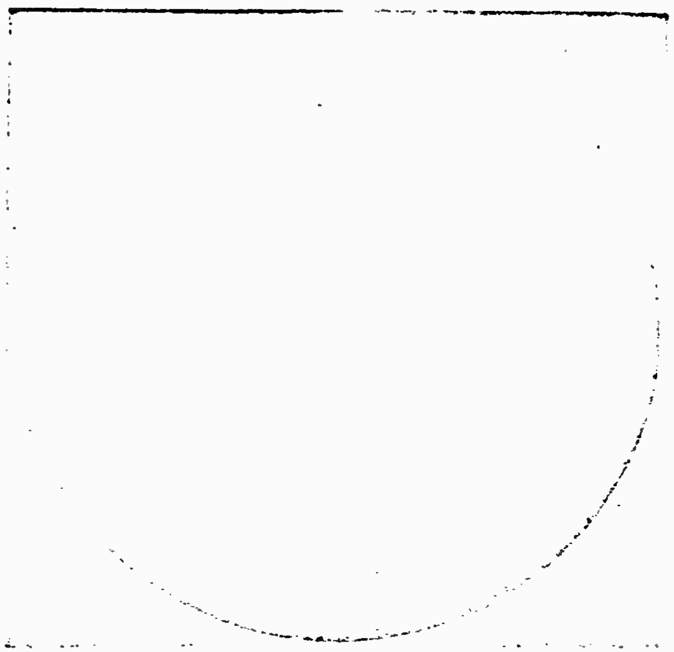
5BT-3A (E) After Etch

θ MAX = 35.5
θ MIN = 9.5
$\Delta\theta$ 26.0

Photo Mag. = 4.5X

CLA = 14.0
RMS = 15.5





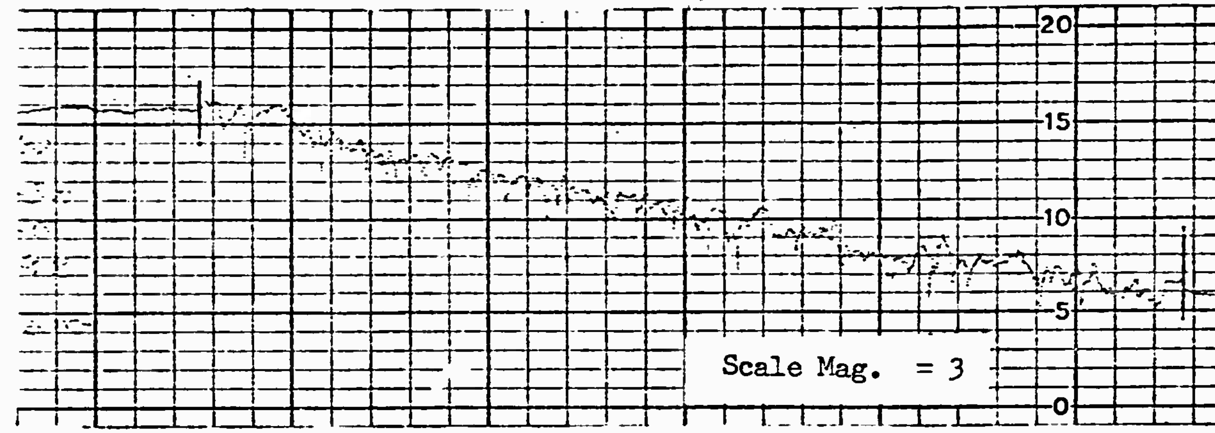


Photo Mag. = 4.4X

CLA = 9.2
RMS = 10.2

7 BT-3A (M) Before Etch

θ MAX	82.5
θ MIN	12.5
$\Delta\theta$	70.0

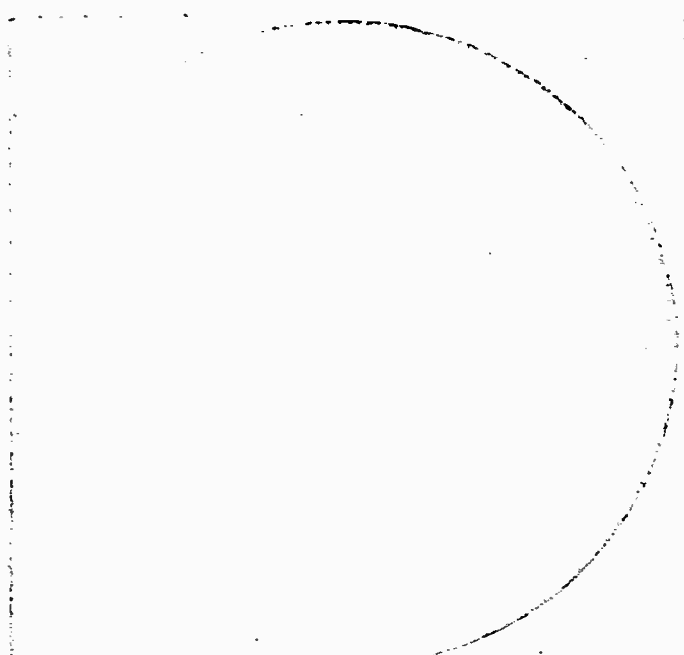
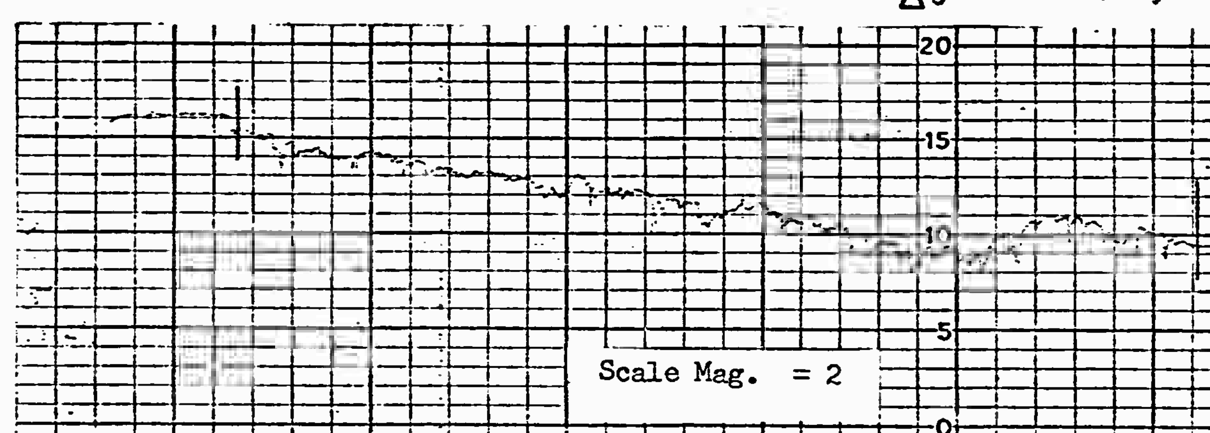


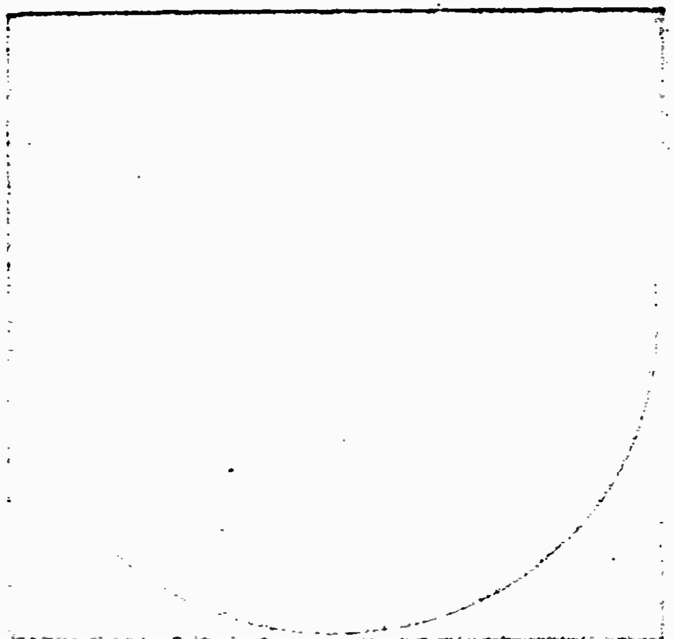
Photo Mag. = 4.5X

CLA = 15.0
RMS = 16.7

7 BT-3A (E) After Etch

θ MAX	52.5
θ MIN	15.5
$\Delta\theta$	37.0





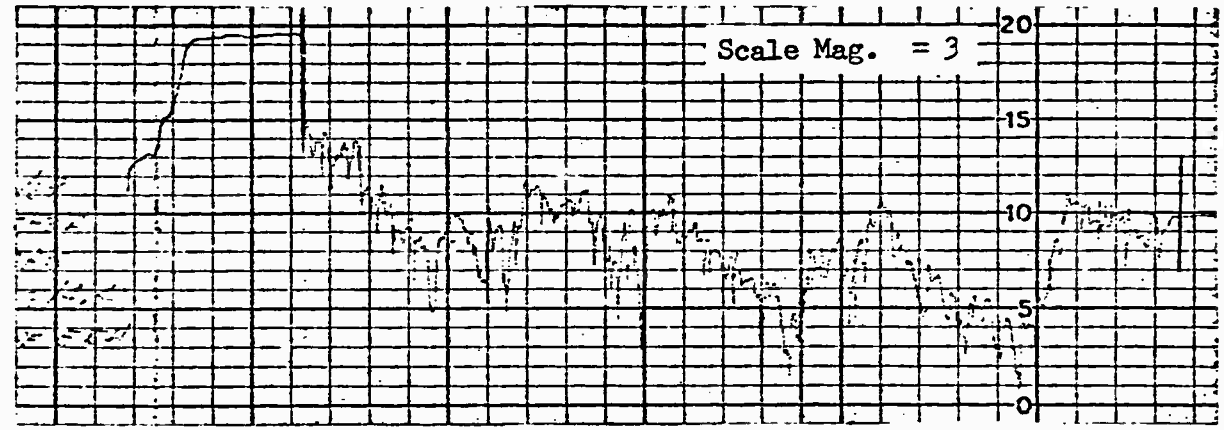
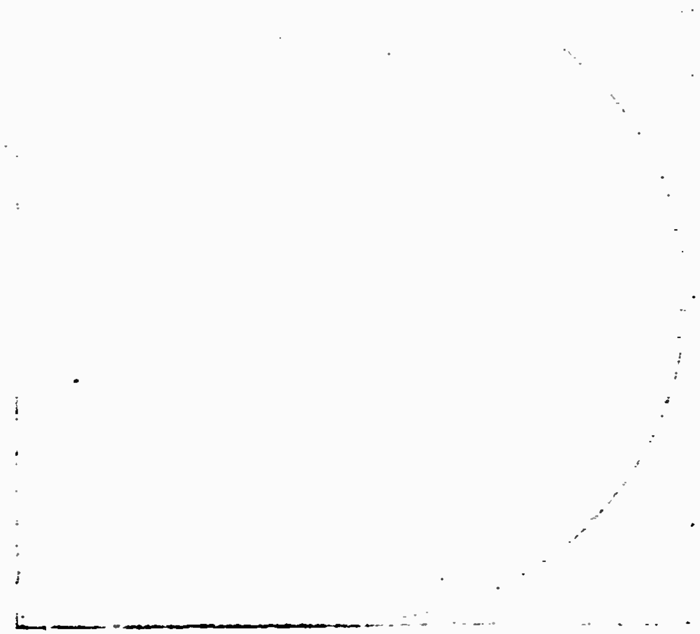


Photo Mag. = 4.4X

CLA = 14.4
RMS = 16.0

10 BT-3A (M) Before Etch

θ MAX	83.5
θ MIN	(9.0)
$\Delta\theta$	74.5

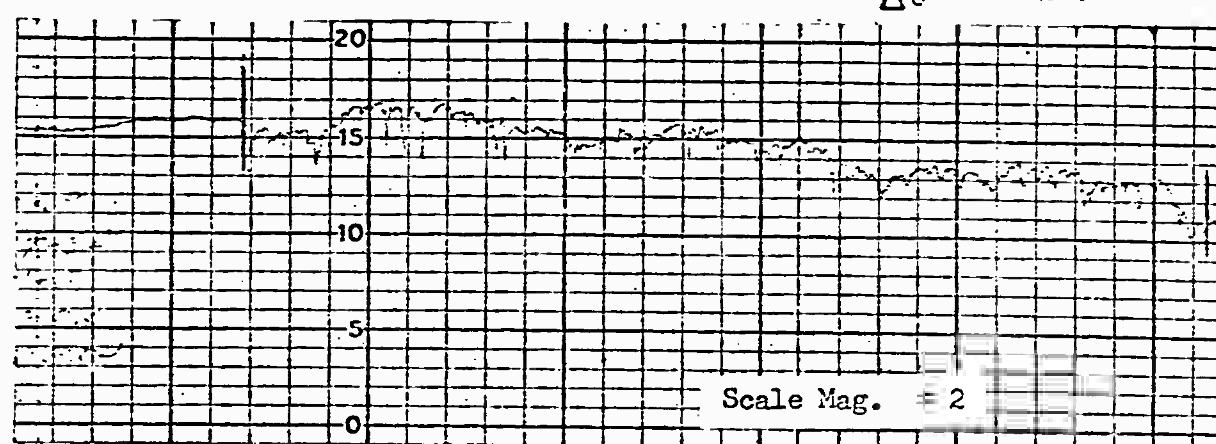


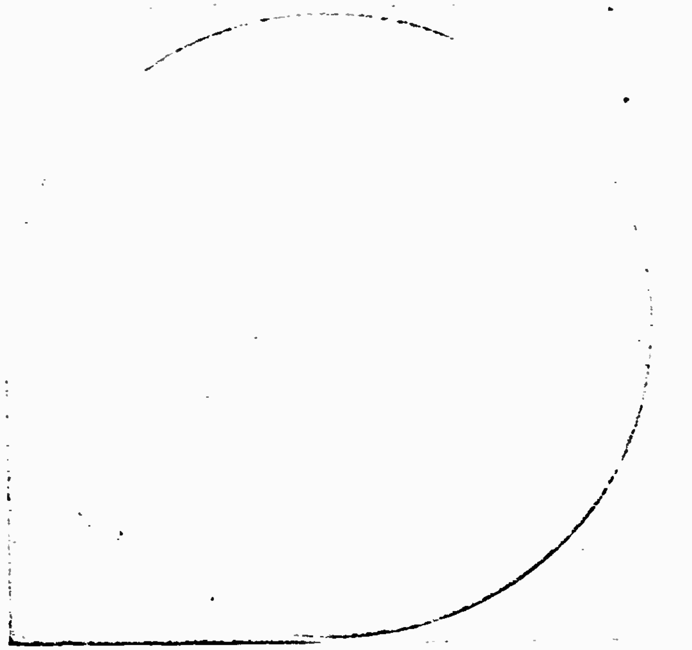
10 BT-3A (E) After Etch

θ MAX	54.0
θ MIN	8.5
$\Delta\theta$	45.5

Photo Mag. = 4.5X

CLA = 24.0
RMS = 26.6





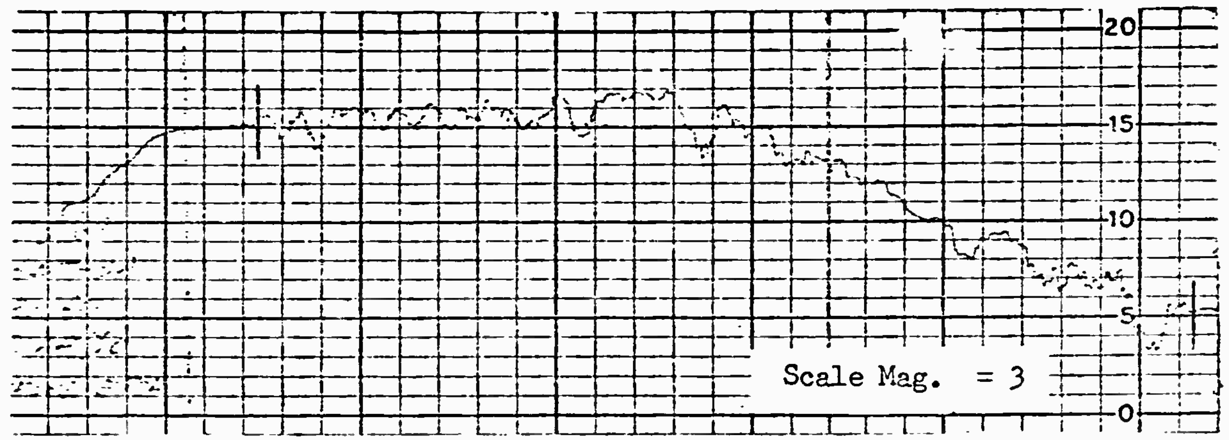


Photo Mag. = 4.4X

CLA = 7.0
RMS = 7.8

20 BT-3A (M) Before Etch

θ MAX	70.0
θ MIN	12.5
$\Delta\theta$	57.5

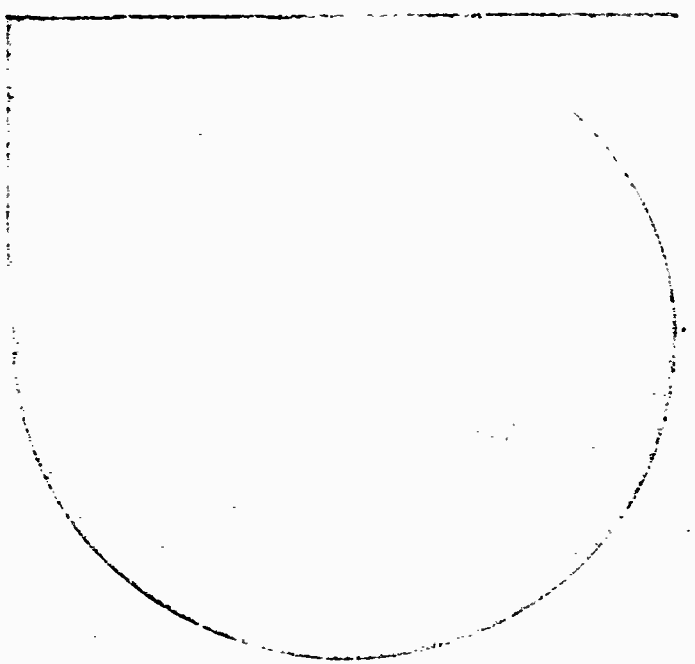
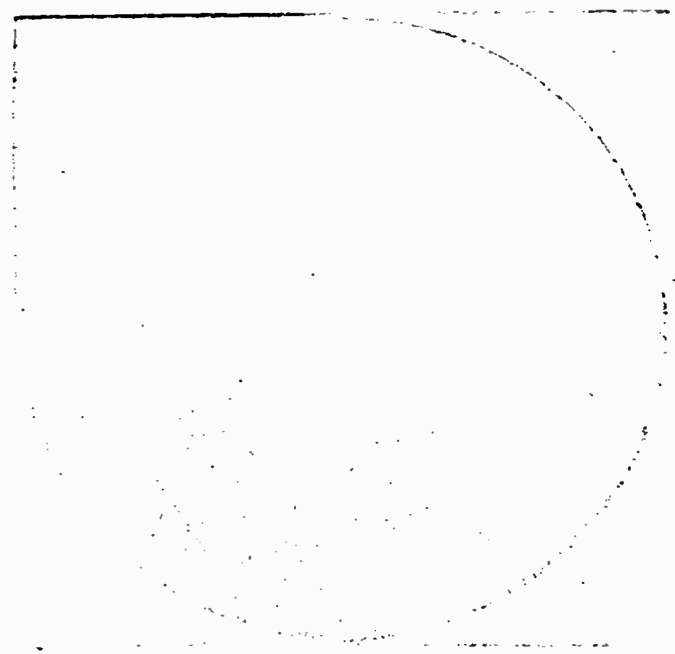


Photo Mag. = 4.5X

CLA = 14.0
RMS = 15.5

20 BT-3A (E) After Etch

θ MAX	36.0
θ MIN	13.5
$\Delta\theta$	22.5



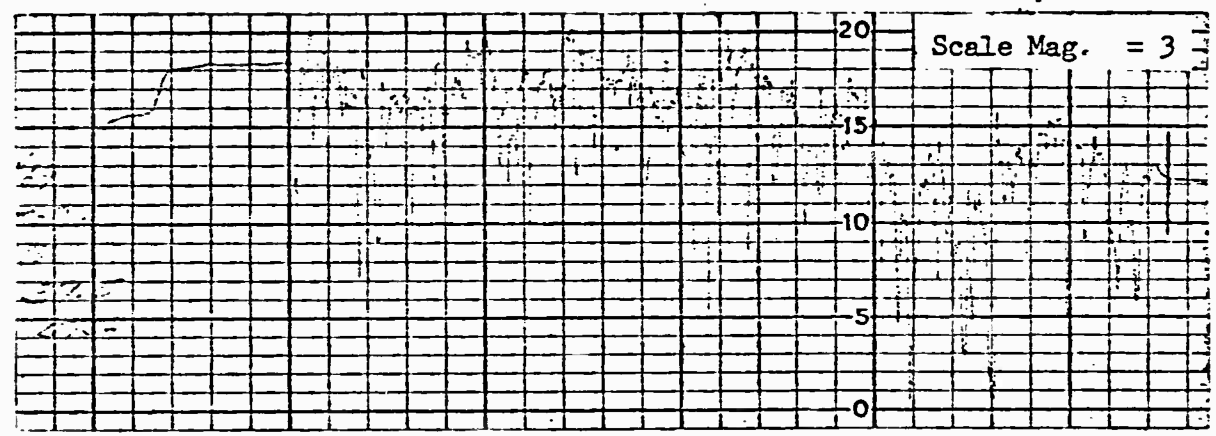


Photo Mag. = 4.4X

CLA = 37.5
RMS = 41.6

40 BT-3A (M) Before Etch

θ MAX	63.5
θ MIN	4.0
$\Delta\theta$	59.5

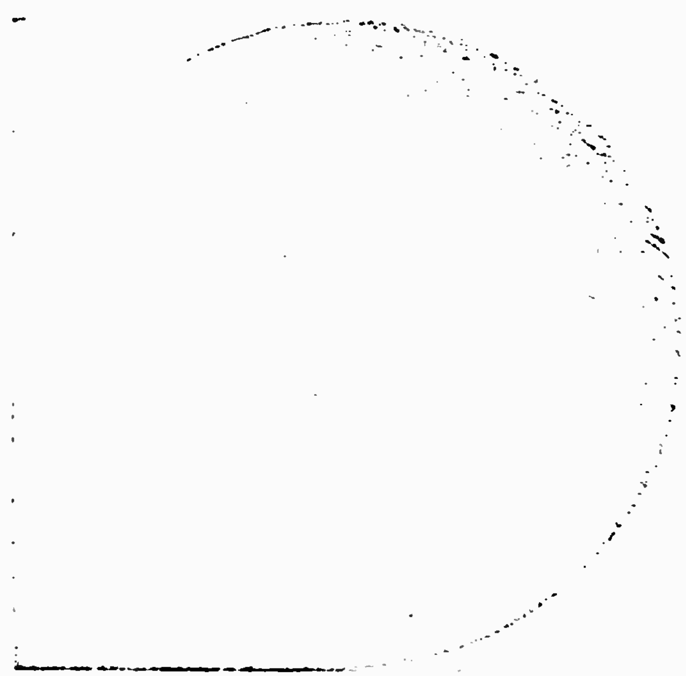
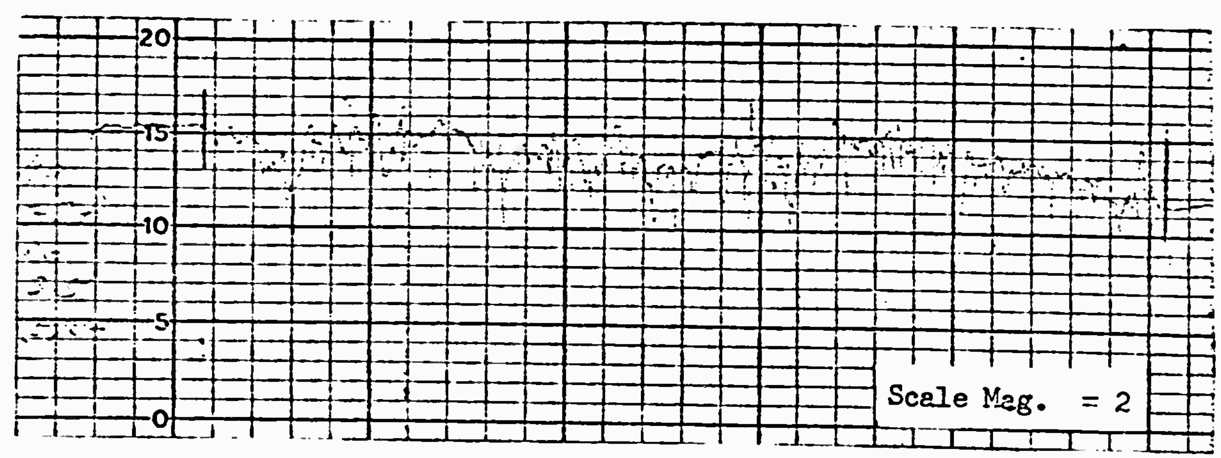


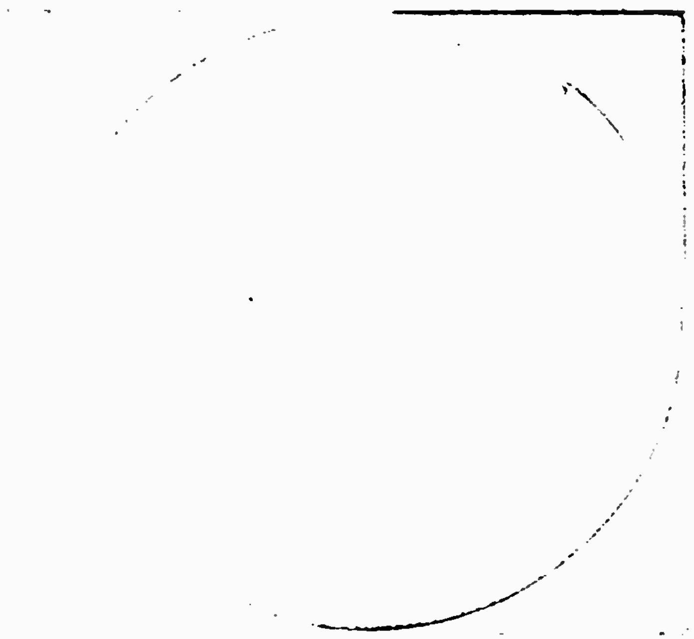
Photo Mag. = 4.5X

CLA = 35.0
RMS = 38.8

40 BT-3A (E) After Etch

θ MAX	19.0
θ MIN	5.0
$\Delta\theta$	14.0





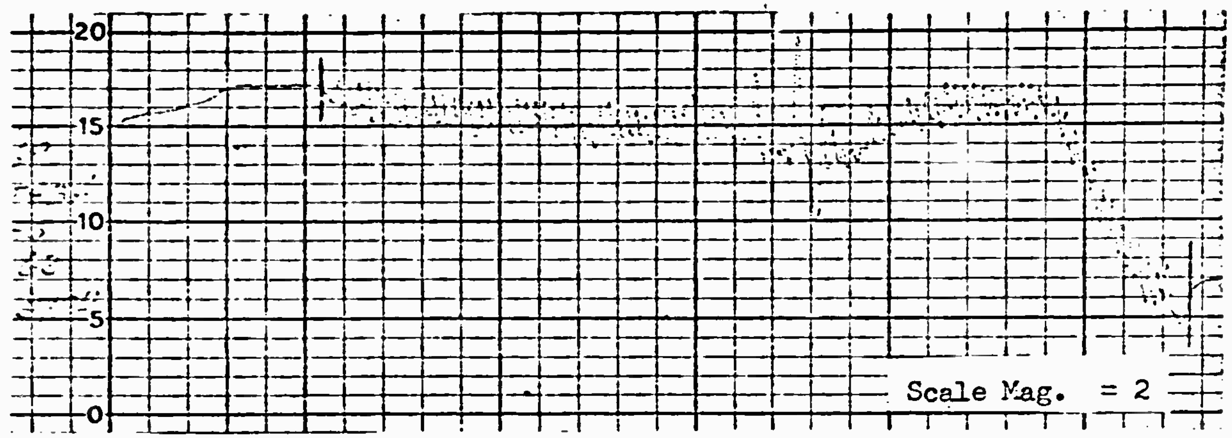


Photo Mag. = 4.4X

CLA = 63
RMS = 69.9

80 BT-3A (M) Before Etch

θ MAX	78.5
θ MIN	12.5
$\Delta\theta$	66.0

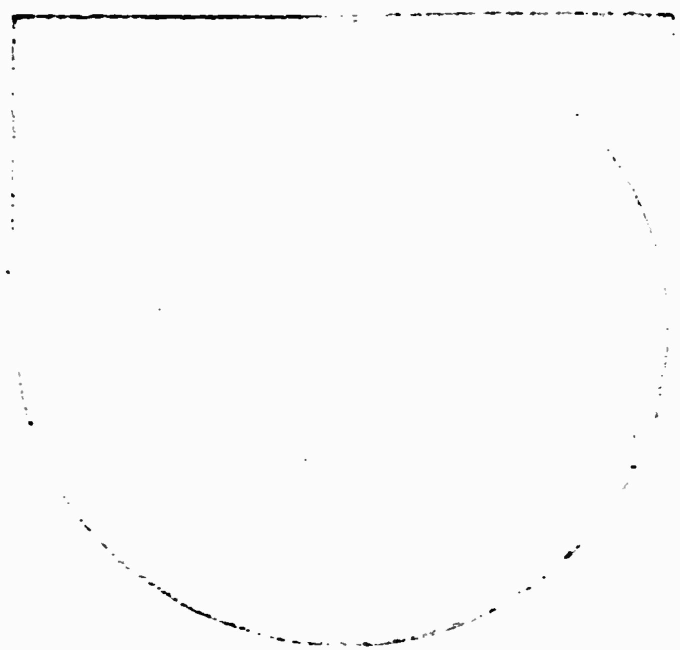
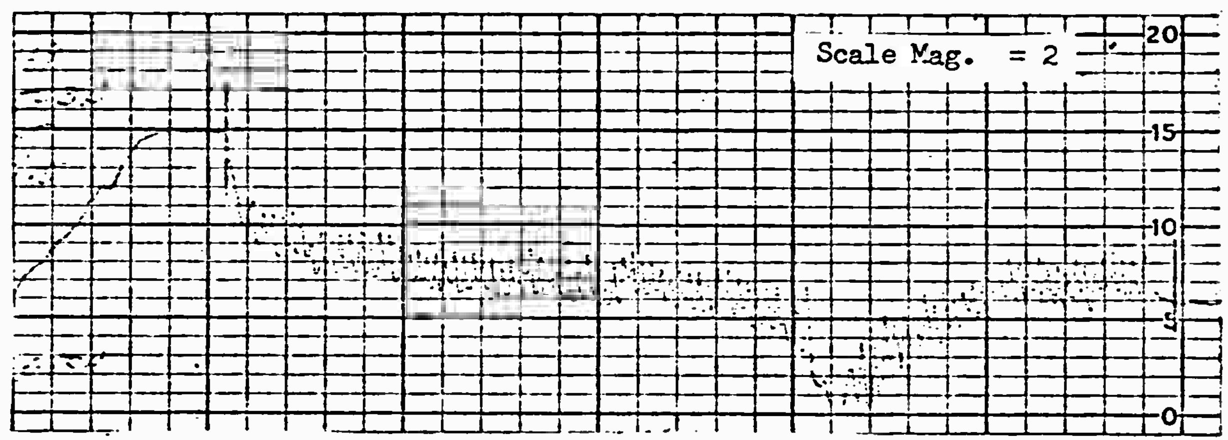


Photo Mag. = 4.5X

CLA = 45.0
RMS = 49.9

80 BT-3A (E) After Etch

θ MAX	43.0
θ MIN	5.5
$\Delta\theta$	37.5



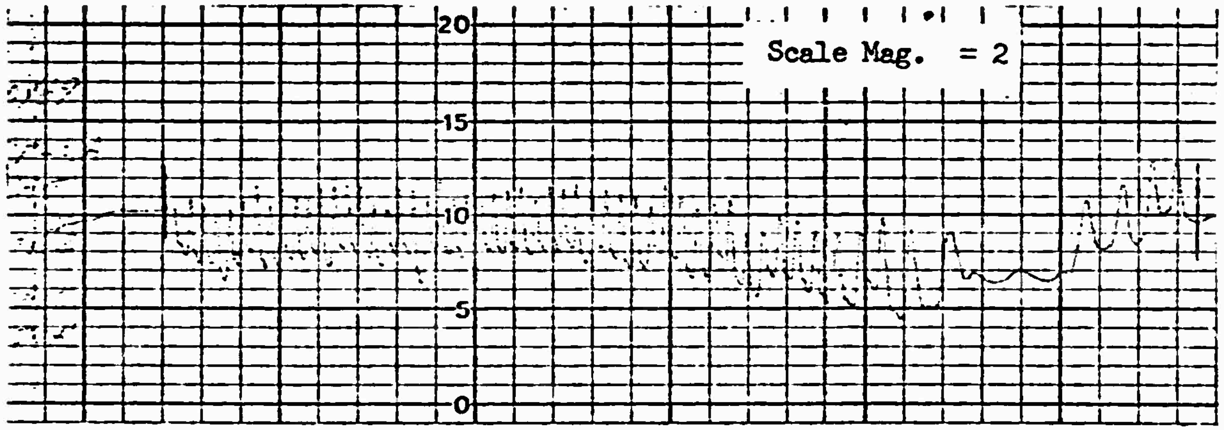


Photo Mag. = 4.4X

CLA = 62
RMS = 68.9

110 BT-3A (M) Before Etch

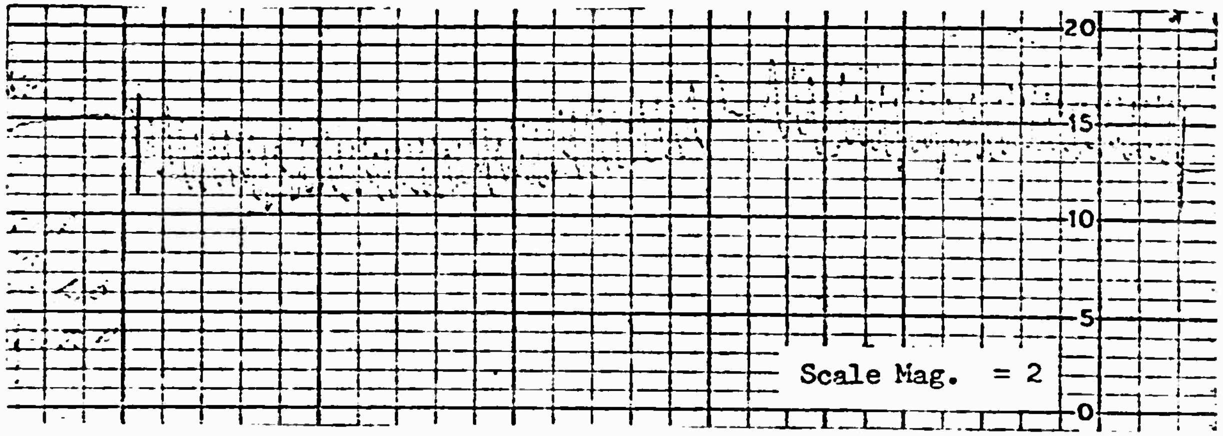
θ MAX	80.5
θ MIN	17.5
$\Delta\theta$	63.0

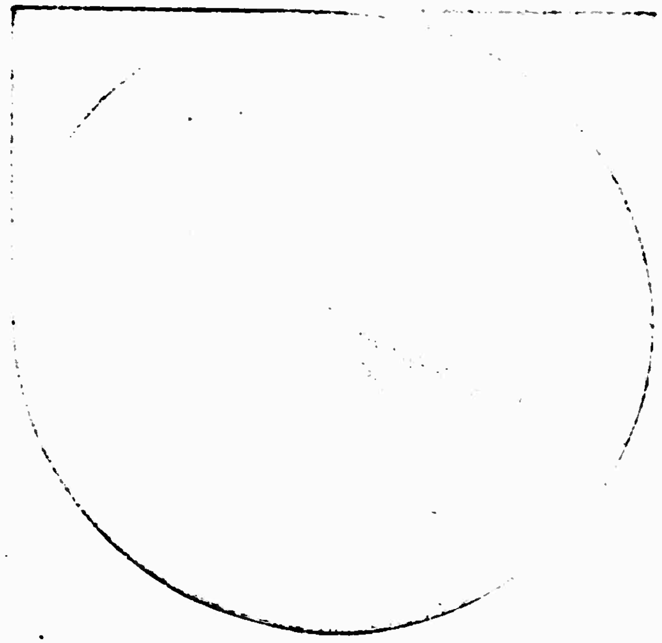
Photo Mag. = 4.5X

CLA = 60.0
RMS = 66.6

110 BT-3A (E) After Etch

θ MAX	32.5
θ MIN	8.0
$\Delta\theta$	24.5





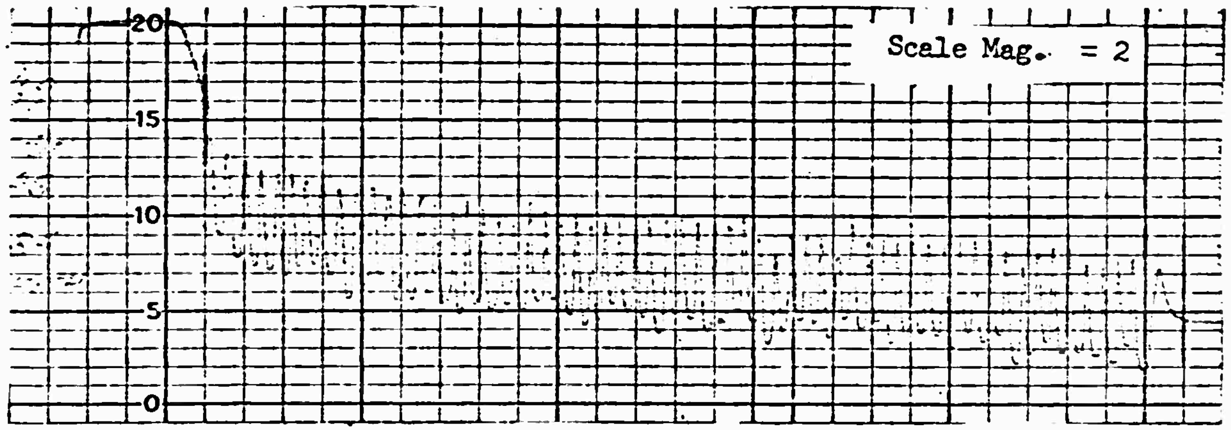


Photo Mag. = 4.4X

CLA = 84
RMS = 93.2

150 BT-3A (M) Before Etch

θ MAX	83.5
θ MIN	27.0
$\Delta\theta$	56.5

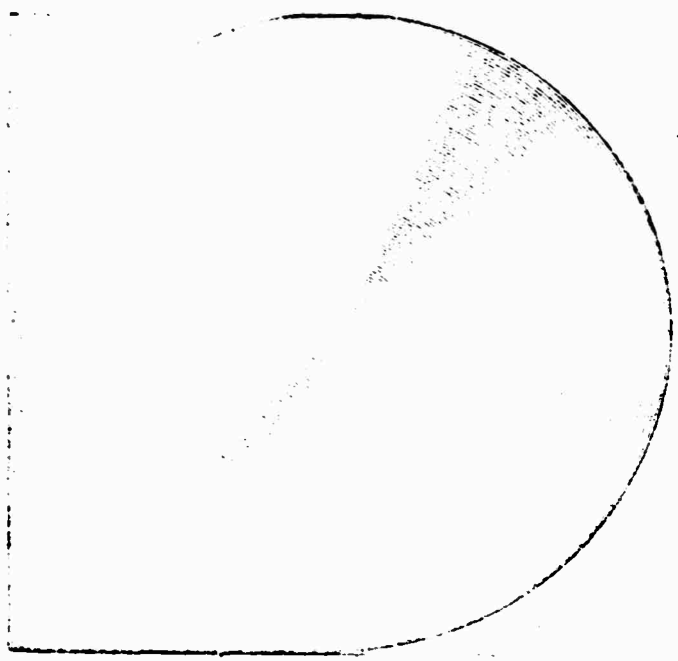
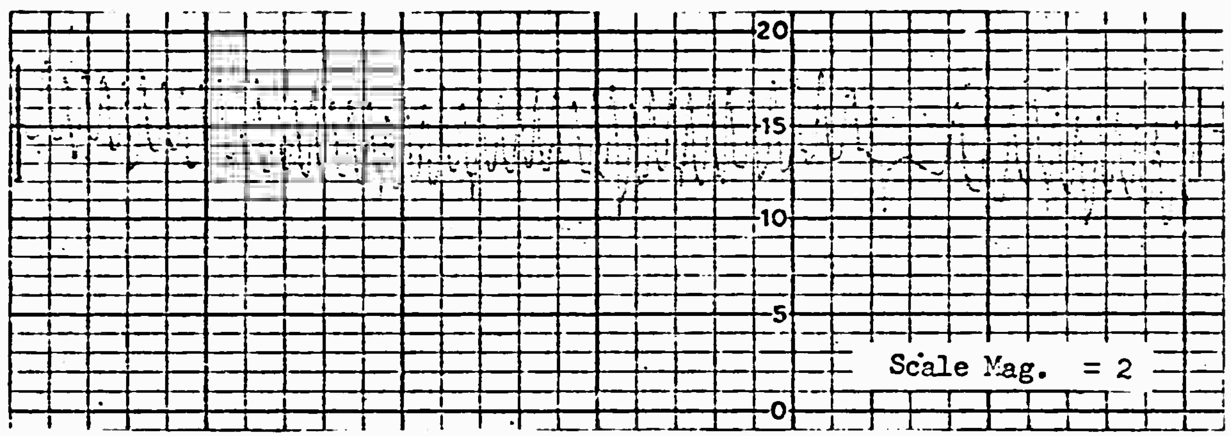


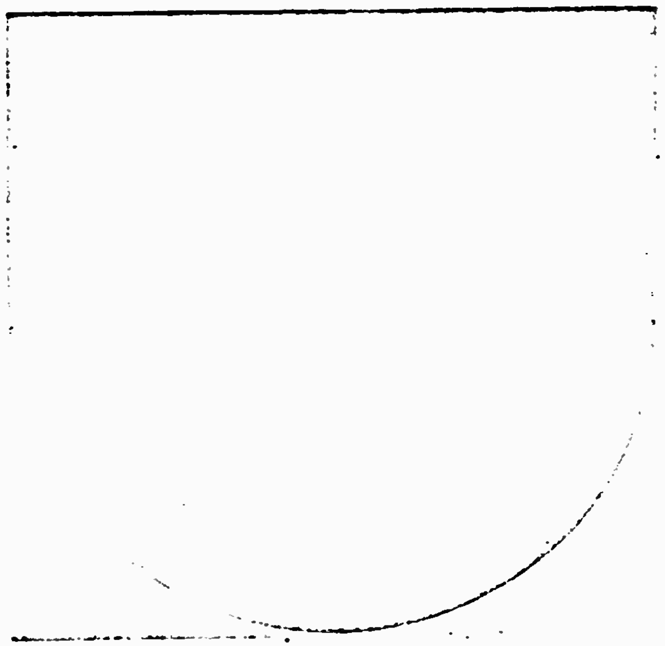
Photo Mag. = 4.5X

CLA = 77.0
RMS = 85.5

150 BT-3A (E) After Etch

θ MAX	46.5
θ MIN	12.0
$\Delta\theta$	34.5





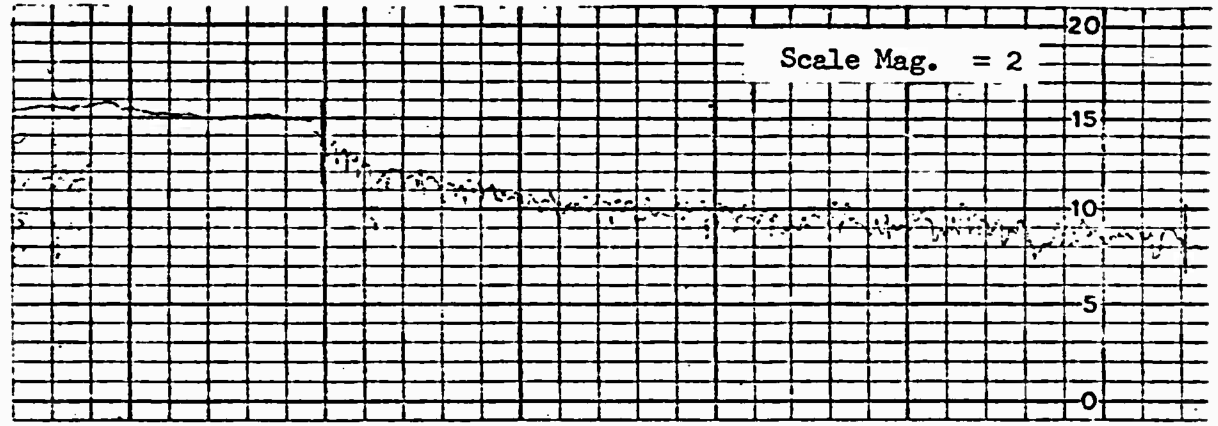


Photo Mag. = 4.4X

CLA = 22
RMS = 24.4

FGLP-BT-3A (M) Before Etc

θ MAX	56.0"
θ MIN	20.0
$\Delta\theta$	36.0

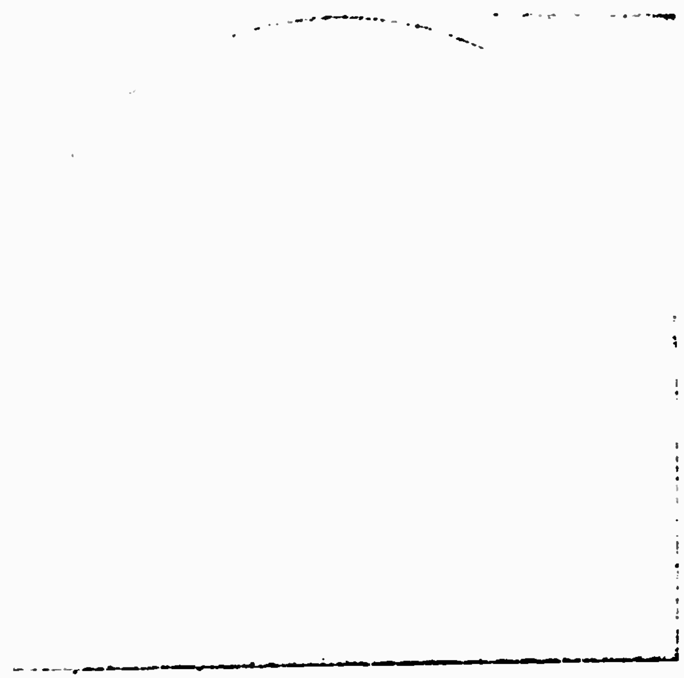
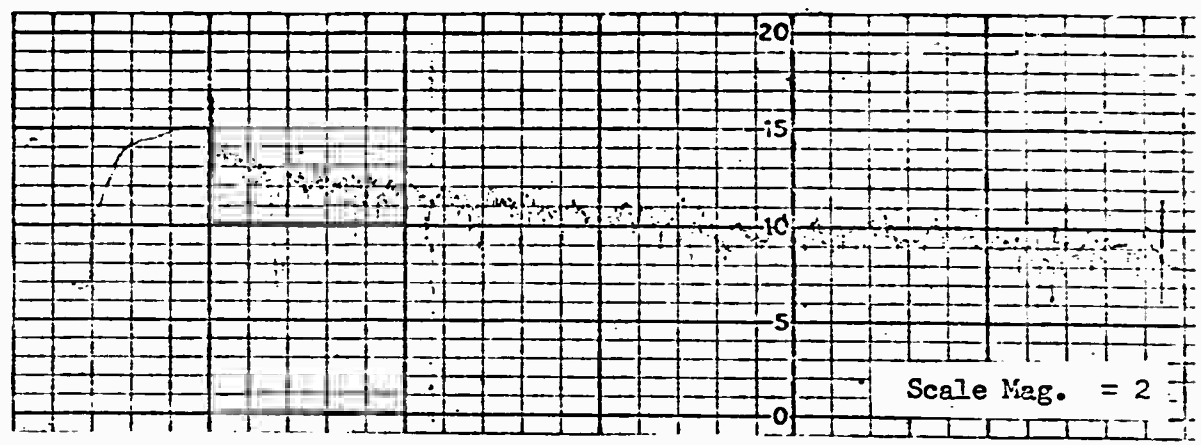


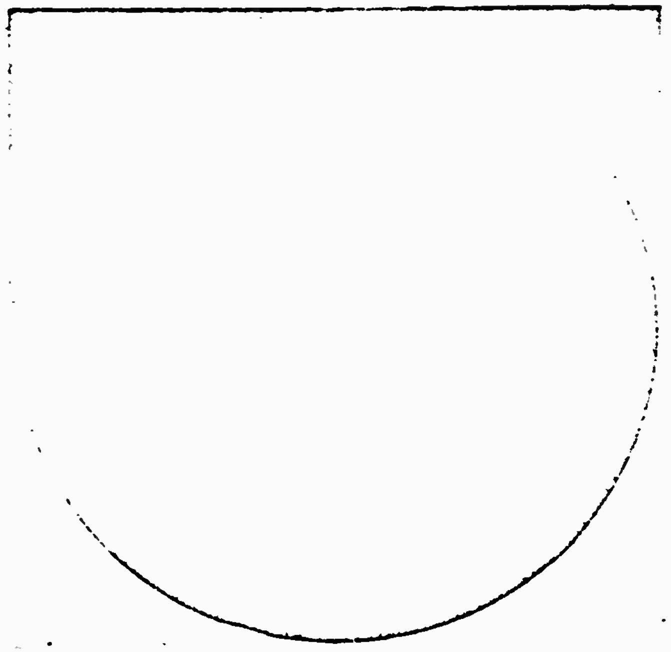
Photo Mag. = 4.5X

CLA = 25.0
RMS = 27.7

FGLP-BT-3A (E) After Etch

θ MAX	44.5
θ MIN	9.5
$\Delta\theta$	35.0





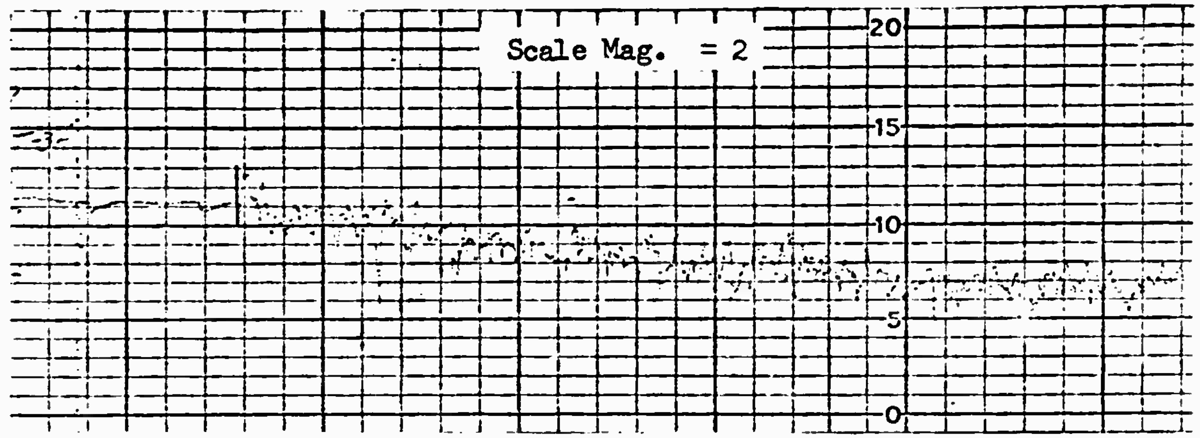


Photo Mag. = 4.4X

CLA = 29
RMS = 32.2

FGHP-BT-3A (M) Before Etc

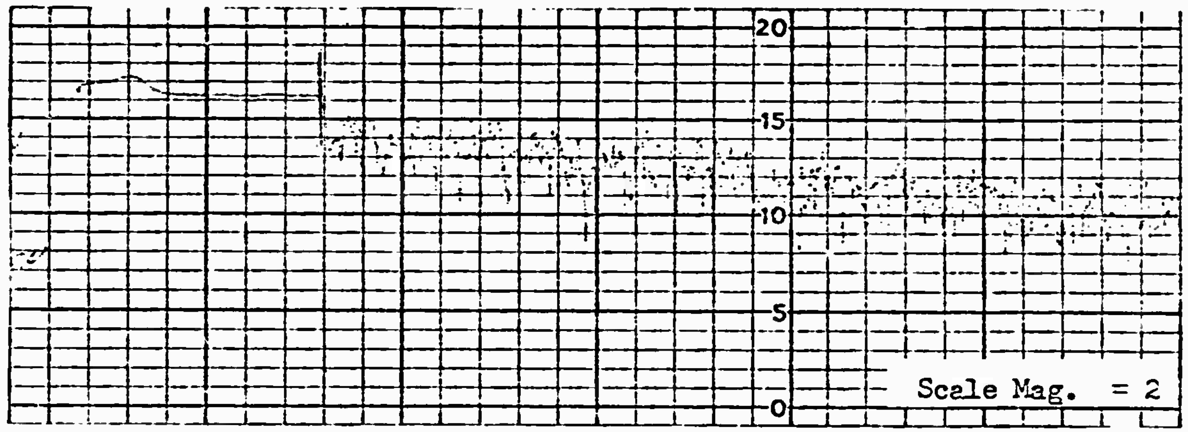
θ MAX 56.0
θ MIN 20.0
$\Delta\theta$ 36.0

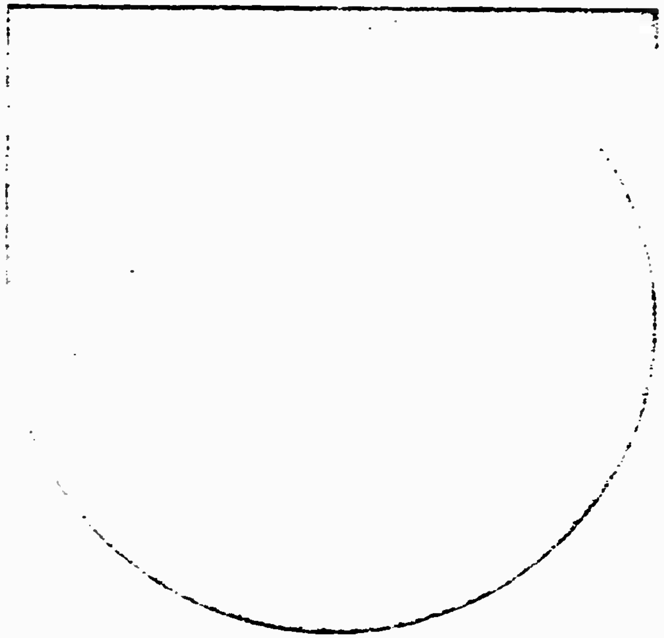
Photo Mag. = 4.5X

CLA = 50.0
RMS = 55.5

FGHP-BT-3A (E) After Etch

θ MAX 59.0
θ MIN (« 31.0)
$\Delta\theta$ —(> 28)





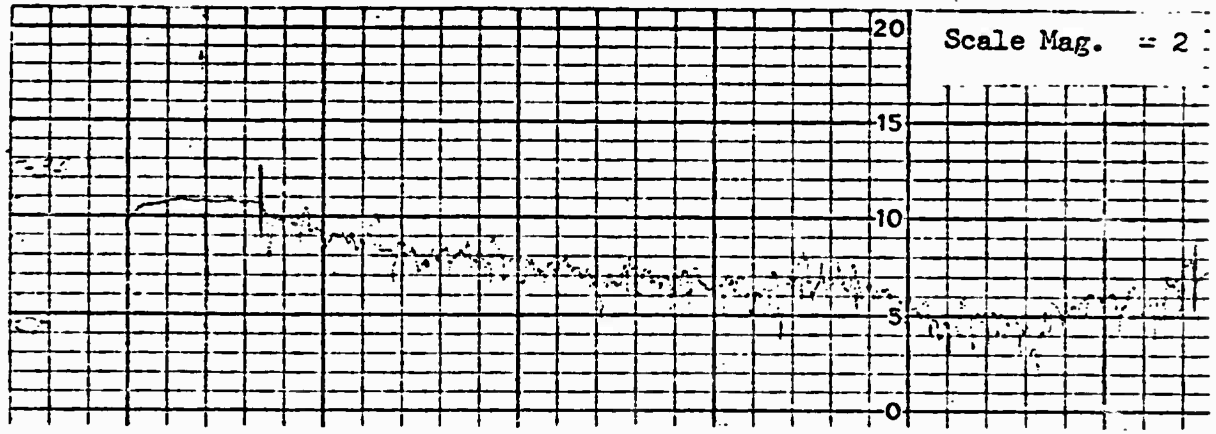
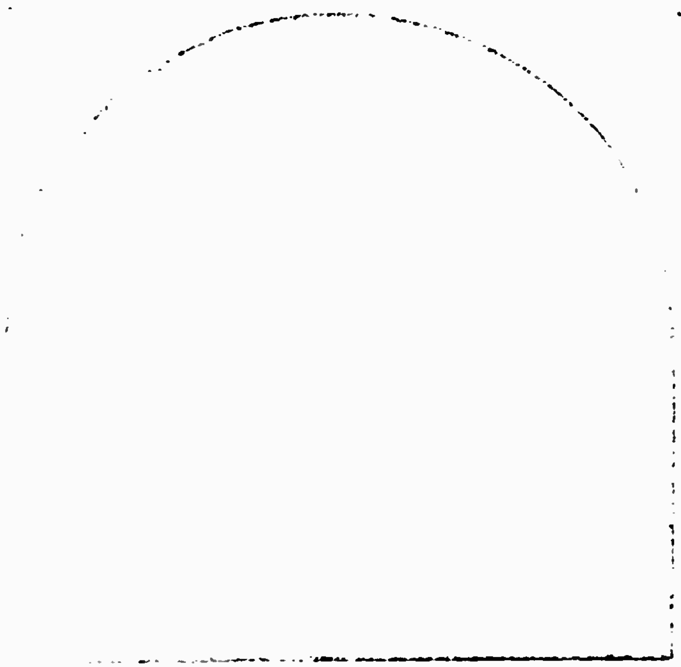


Photo Mag. = 4.4X

CLA = 31.0
RMS = 34.4

CGLP-BT-3A (M) Before Etch

θ MAX	76.0
θ MIN	15.5
$\Delta\theta$	60.5

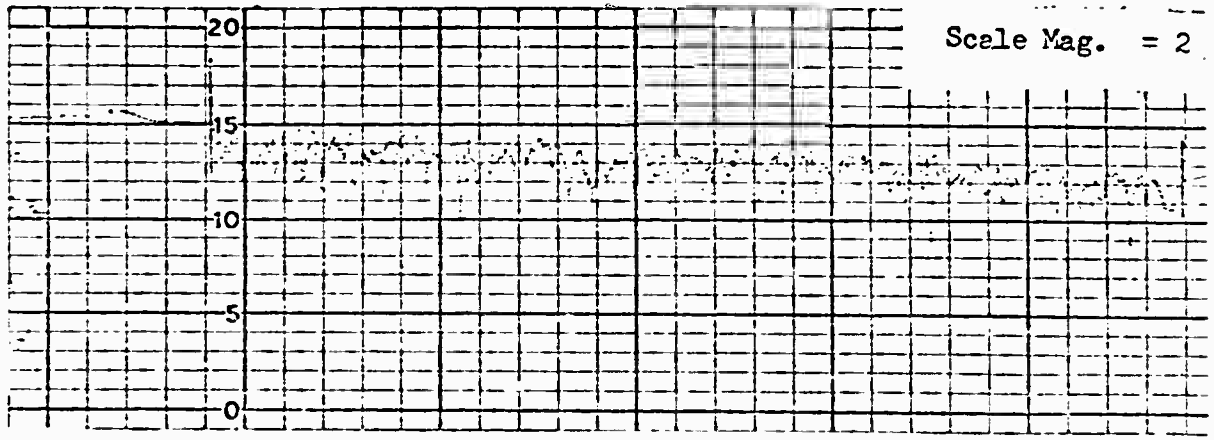


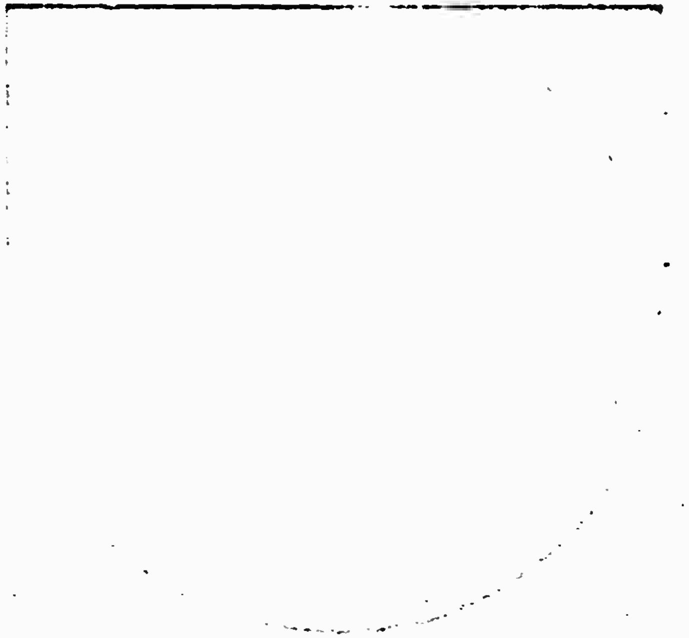
CGLP-BT-3A (E) After Etch

θ MAX	54.5
θ MIN	10.5
$\Delta\theta$	44.0

Photo Mag. = 4.5X

CLA = 30.0
RMS = 33.3





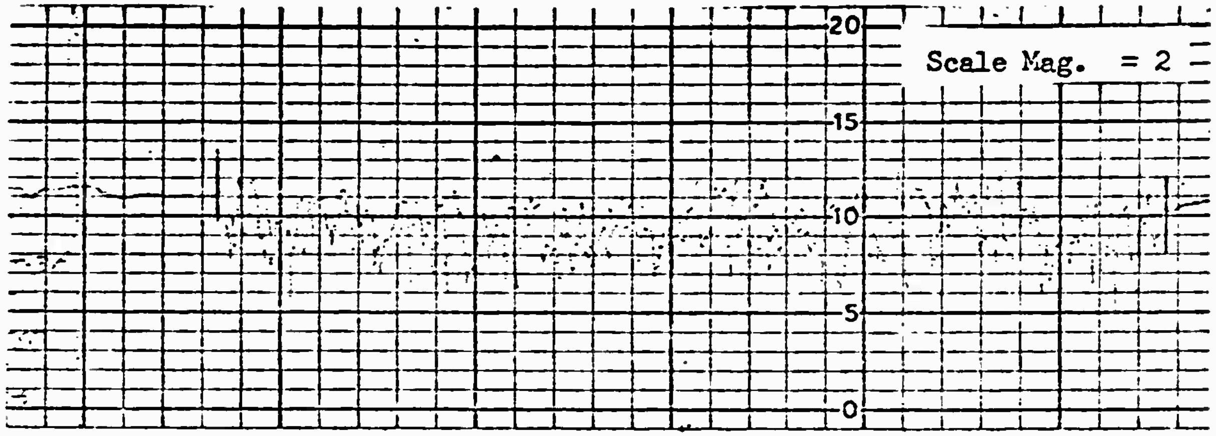


Photo Mag. = 4.4X

CLA = 55.0
RMS = 61.0

CGHP-BT-3A (M) Before Etch

θ MAX	78.0
θ MIN	13.0
$\Delta\theta$	65.0

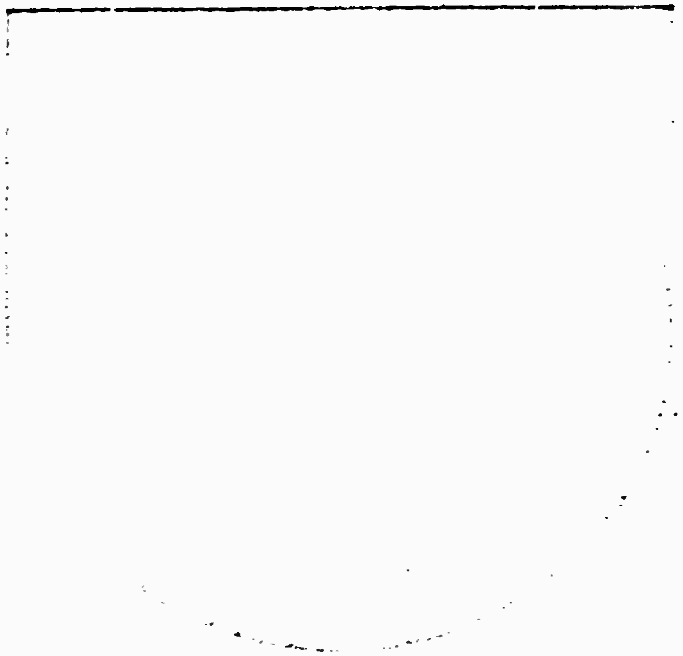
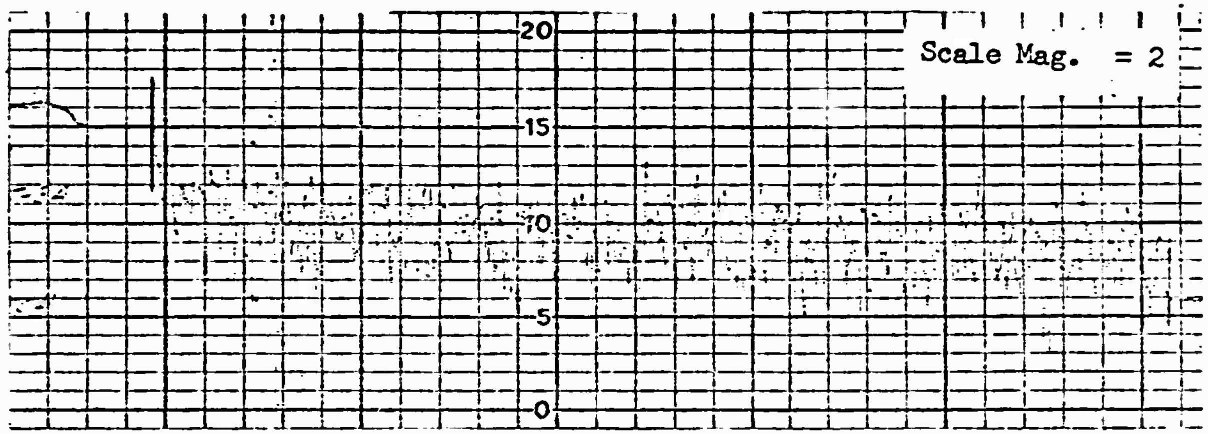


Photo Mag. = 4.5X

CLA = 70.0
RMS = 77.7

CGHP-BT-3A (E) After Etch

θ MAX	40.5
θ MIN	9.0
$\Delta\theta$	31.5



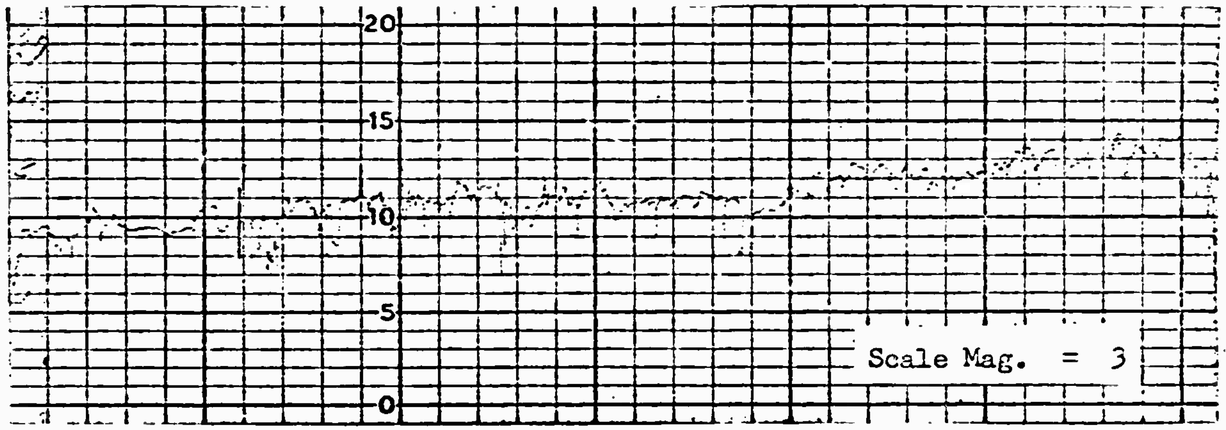


Photo Mag. = 4.4X

CLA = 2.2
RMS = 2.4

1 CS-3A (M) Before Etch

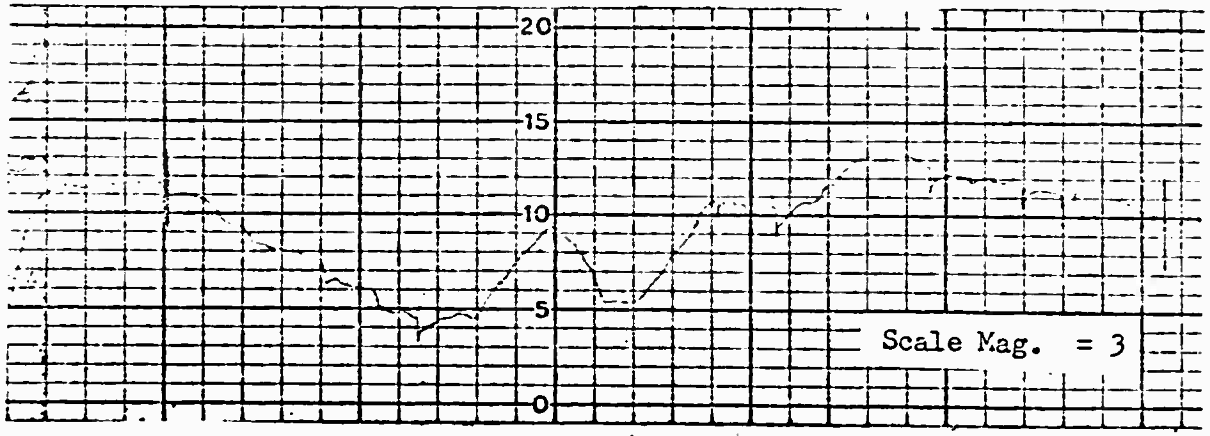
θ MAX	88.0
θ MIN	44.5
$\Delta\theta$	43.5

Photo Mag. = 4.7X

CLA = 2.5
RMS = 2.8

1 CS-3A (E) After Etch

θ MAX	24.5
θ MIN	4.5
$\Delta\theta$	20.0



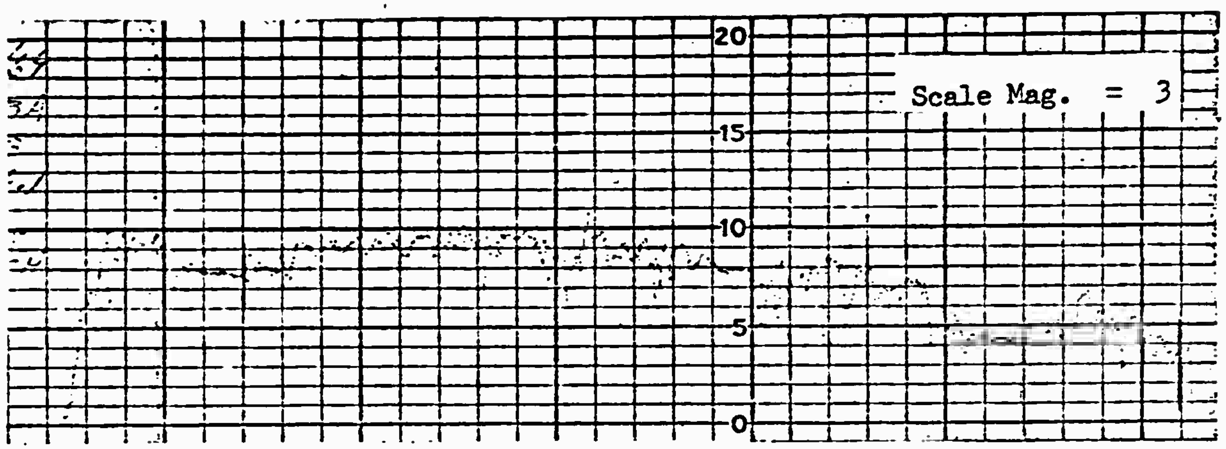


Photo mag. = 4.4X

CLA = 4.1
RMS = 4.5

5 CS-3A (M) Before Etch

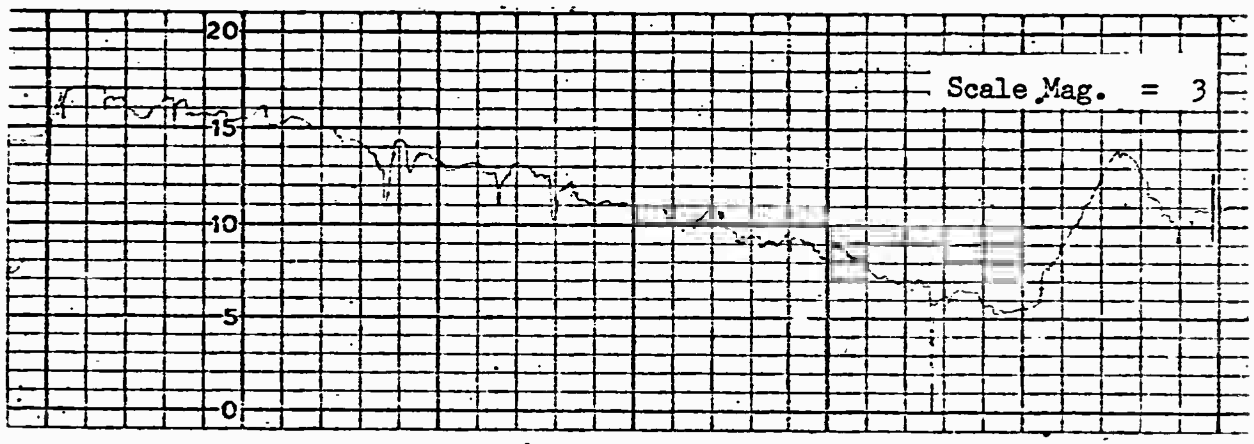
θ MAX	74.0
θ MIN	15.0
$\Delta\theta$	59.0

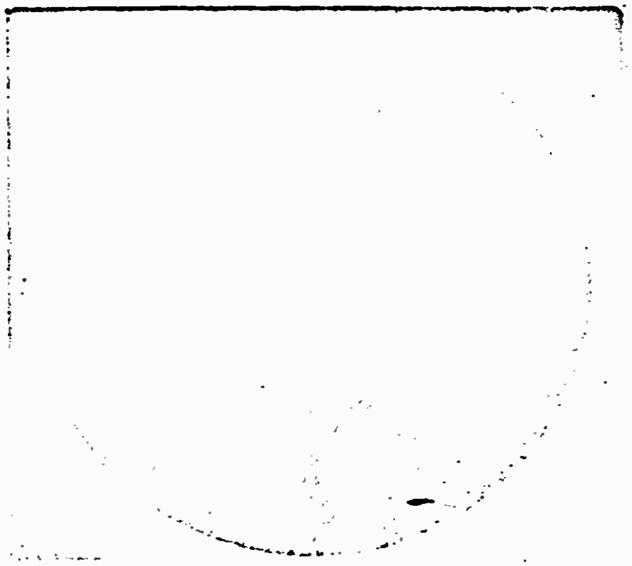
Photo Mag. = 4.5X

CLA = 6.5
RMS = 7.2

5 CS-3A (E) After Etch

θ MAX	15.0
θ MIN	4.5
$\Delta\theta$	10.5





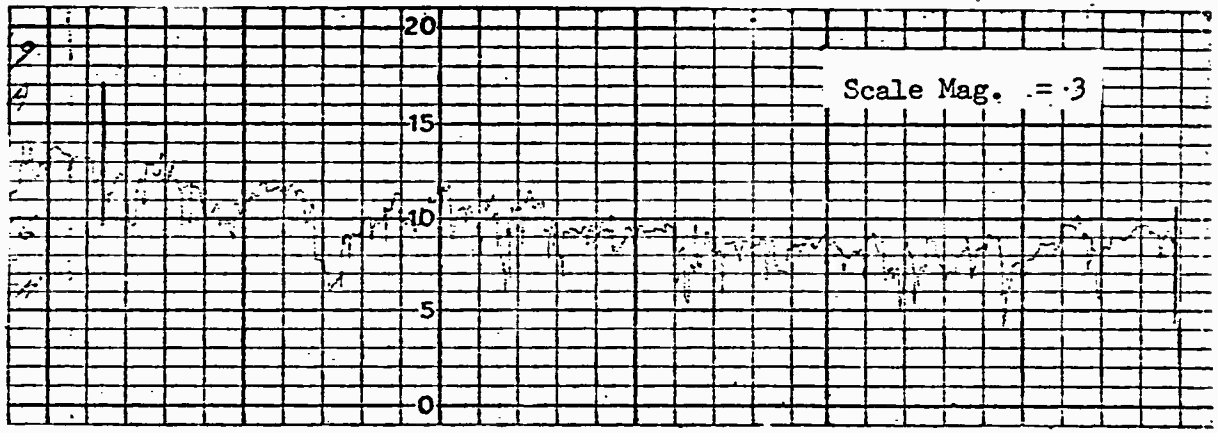


Photo mag. = 3.7X

CLA = 9.6
RMS = 10.6

7CS-3A (M) Before Etch

θ MAX	81.0
θ MIN	15.5
$\Delta\theta$	65.5

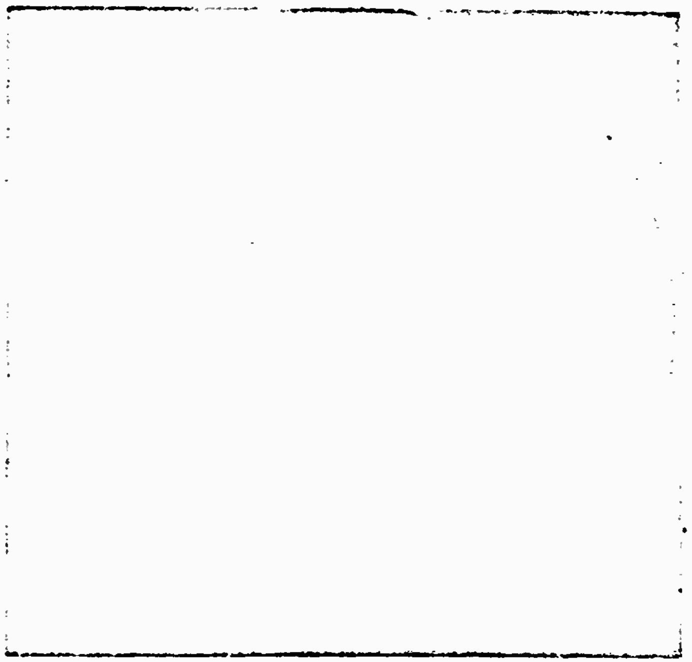
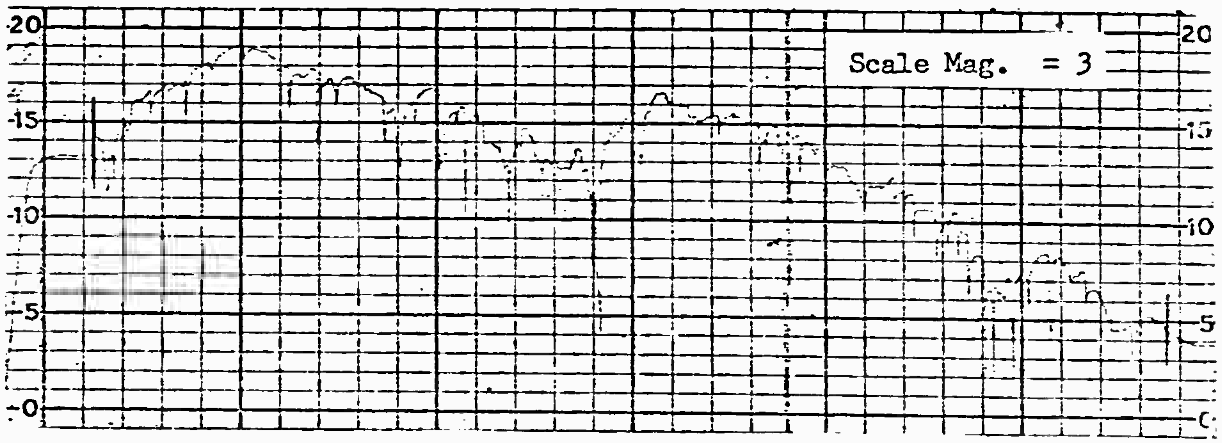


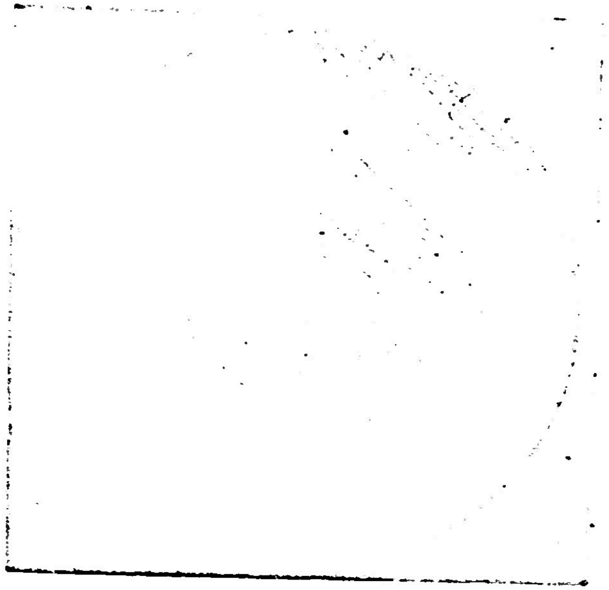
Photo Mag. = 4.5X

CLA = 18.0
RMS = 20.0

7 CS-3A (E) After Etch

θ MAX	18.0
θ MIN	5.0
$\Delta\theta$	13.0





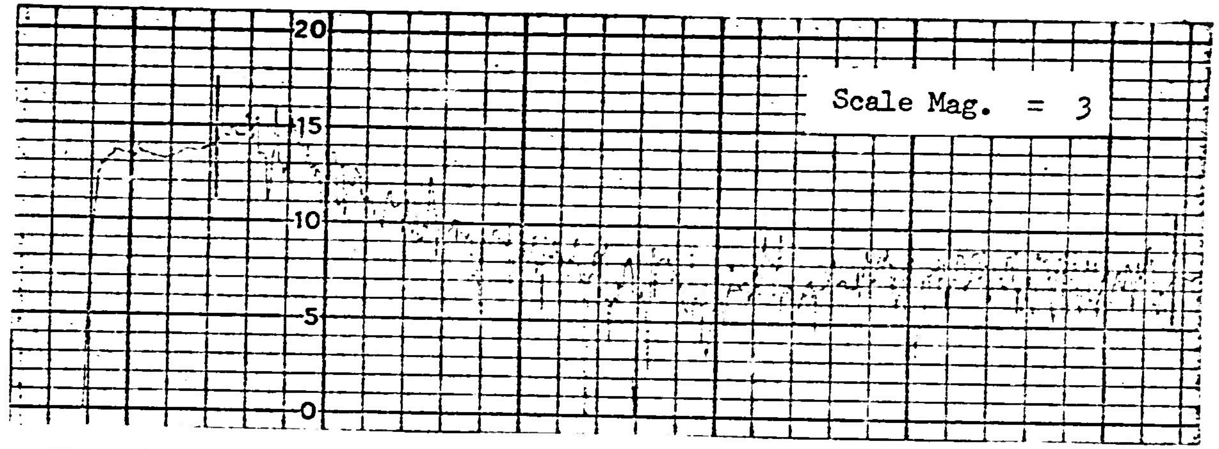


Photo Mag. = 3.7X

CLA = 8.6
RMS = 9.5

10 CS-3A (M) Before Etch

θ MAX	72.0
θ MIN	16.0
$\Delta\theta$	56.0

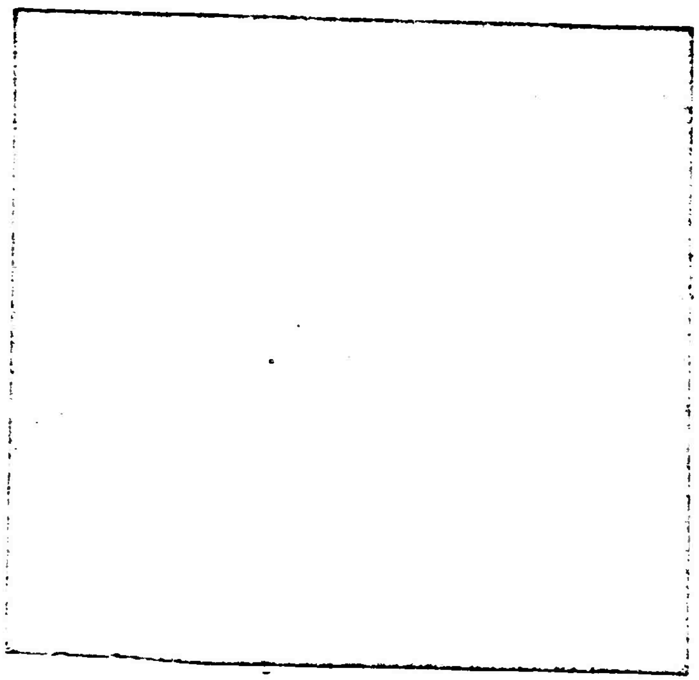
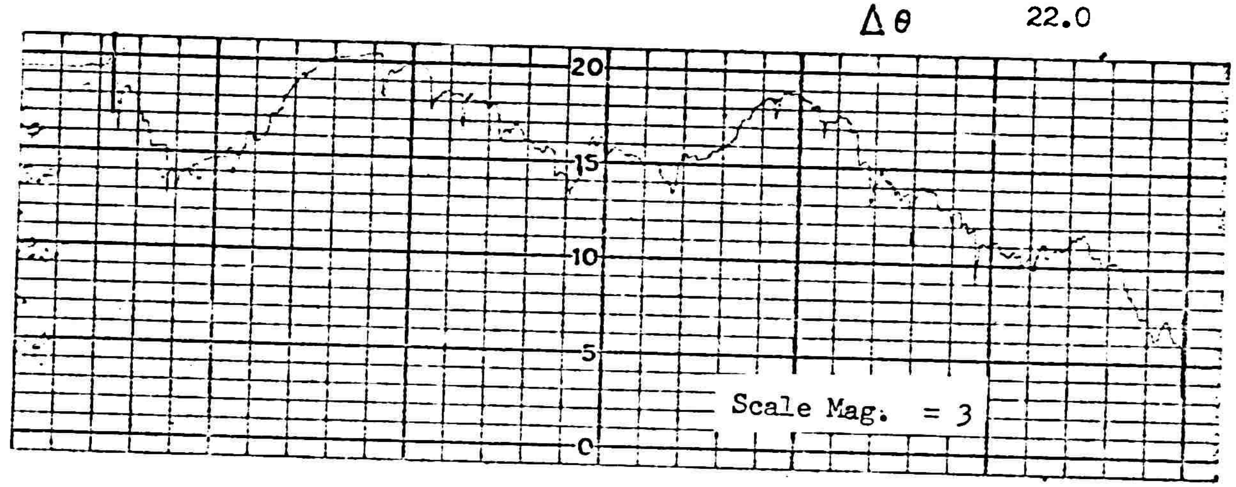


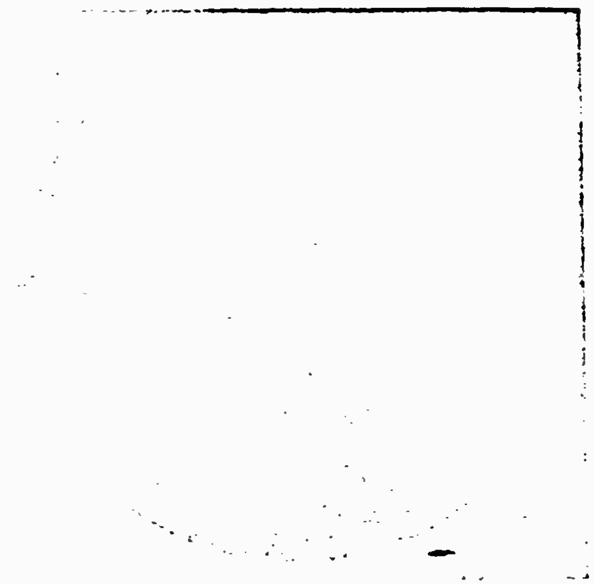
Photo Mag. = 4.5X

CLA = 12.5
RMS = 13.9

10 CS-3A (E) After Etch

θ MAX	28.0
θ MIN	6.0
$\Delta\theta$	22.0





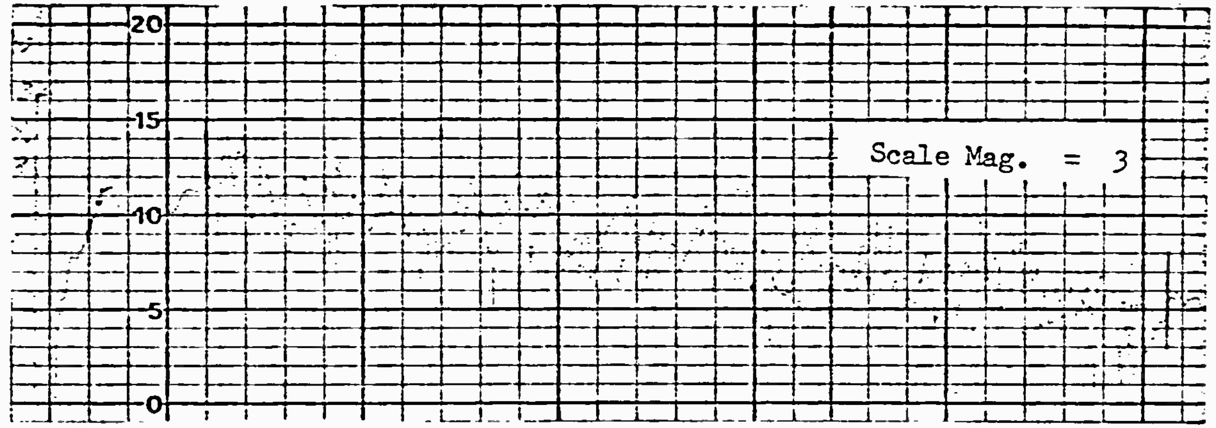


Photo Mag. = 3.7X

CLA = 23.0
RMS = 25.5

20 CS 3A (M) Before Etch

θ MAX	73.5
θ MIN	14.0
$\Delta\theta$	59.5

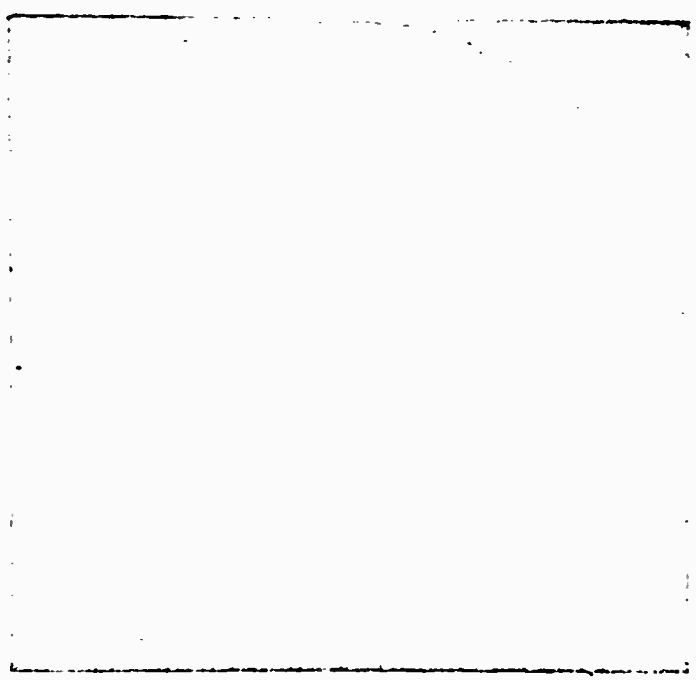
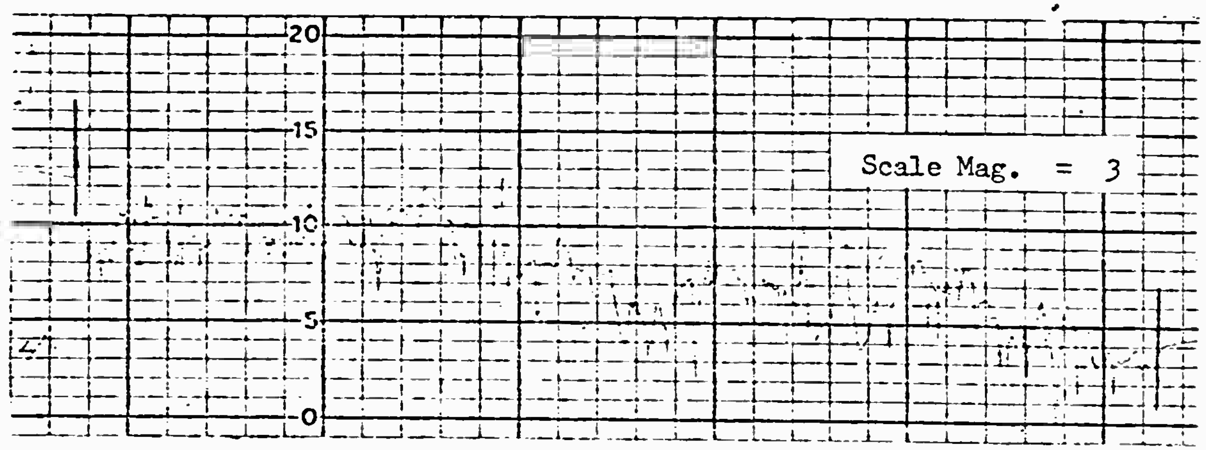


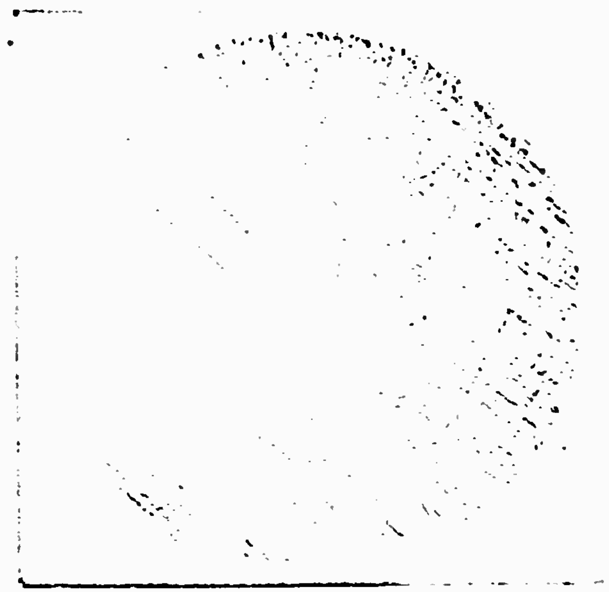
Photo Mag. = 4.5X

CLA = 17.0
RMS = 18.9

20 CS-3A (E) After Etch

θ MAX	28.5
θ MIN	6.0
$\Delta\theta$	22.5





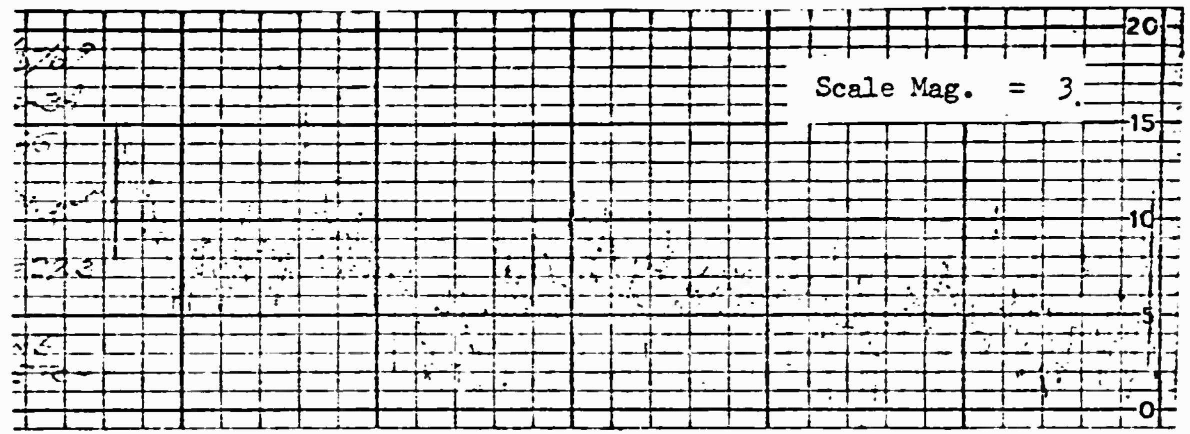


Photo Mag. = 3.7X

CLA = 27.3
RMS = 30.3

40 CS-3A (M) Before Etch

θ MAX 76.0
θ MIN 12.5
$\Delta\theta$ 63.5

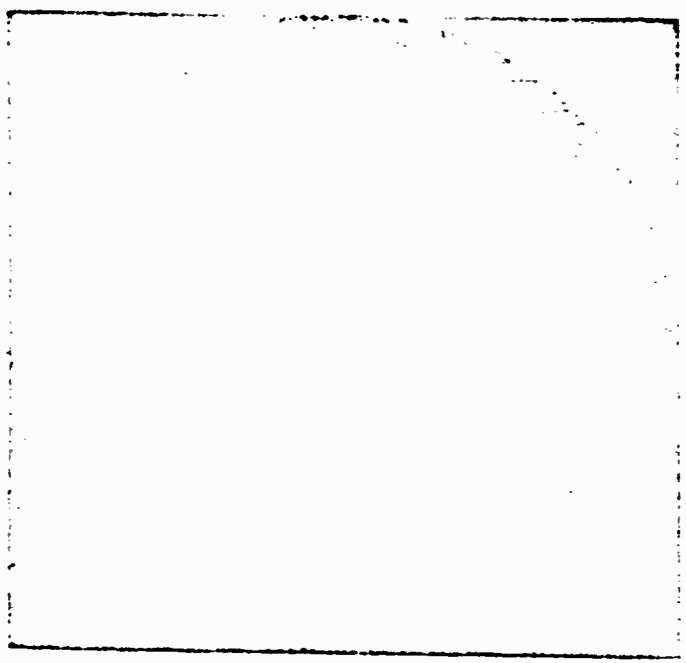
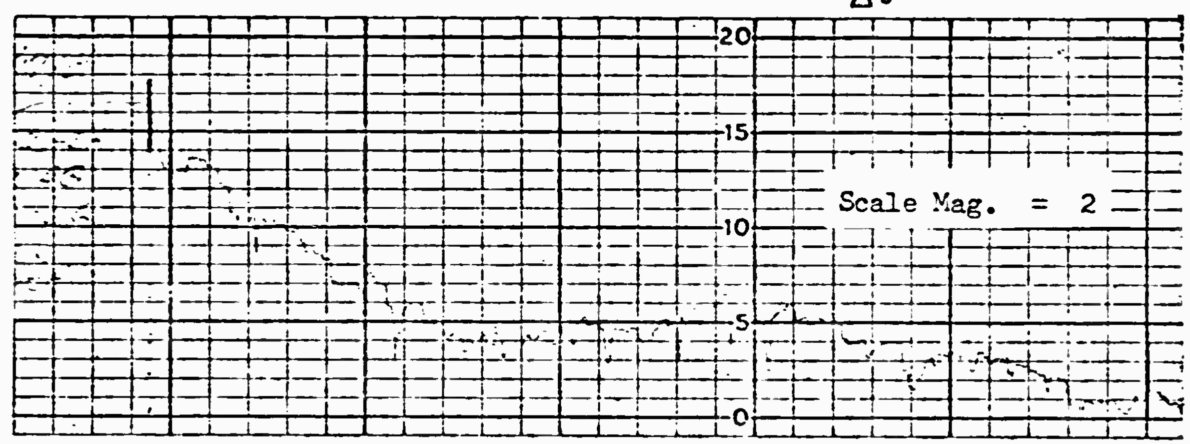


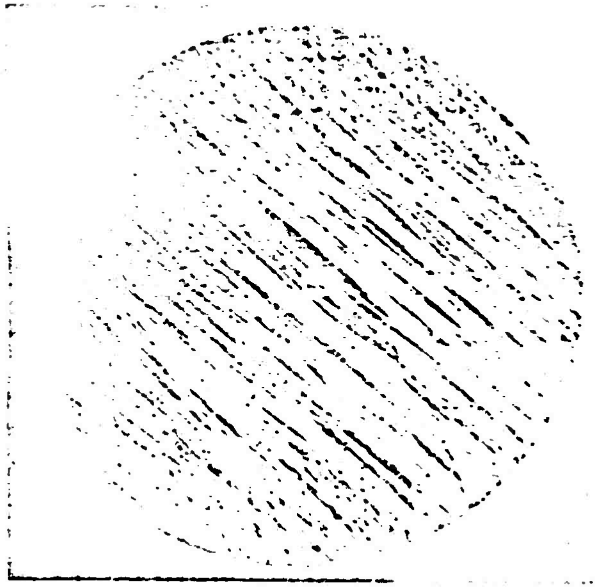
Photo Mag. = 4.5X

CLA = 26.0
RMS = 28.8

40 CS-3A (E) After Etch

θ MAX 25.0
θ MIN (6.5)
$\Delta\theta$ (18.5)





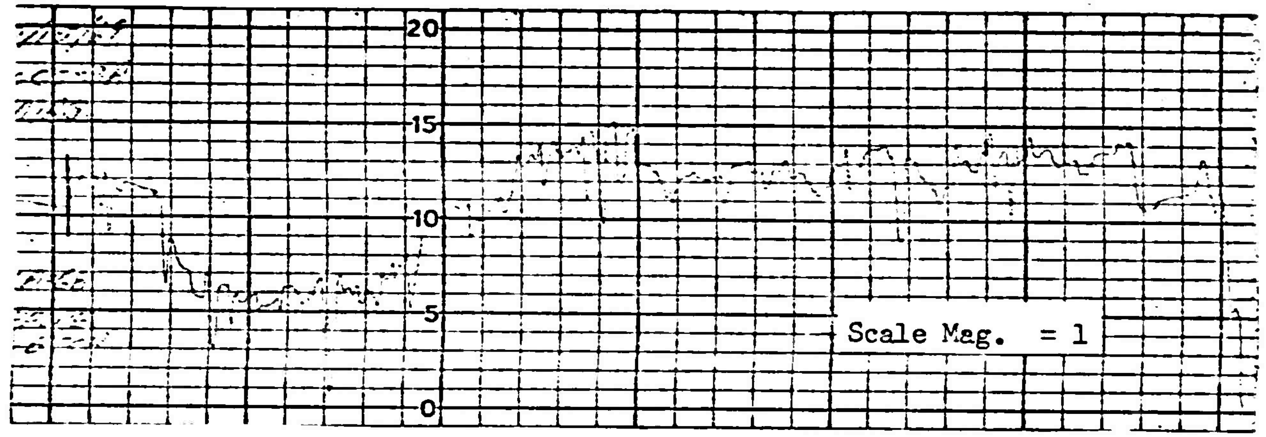


Photo Mag. = 3.7X

CLA = 68.0
RMS = 75.5

80 CS-3A (M) Before Etch

θ MAX	77.5
θ MIN	11.5
$\Delta\theta$	66.0

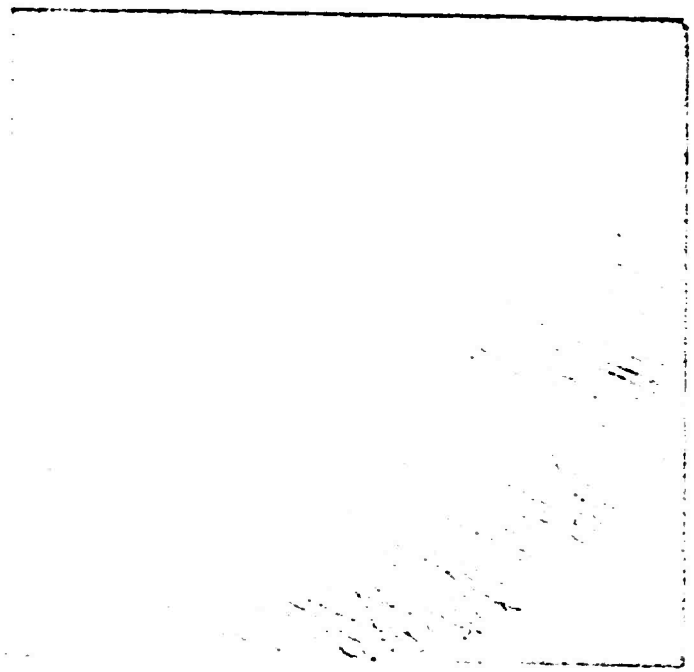
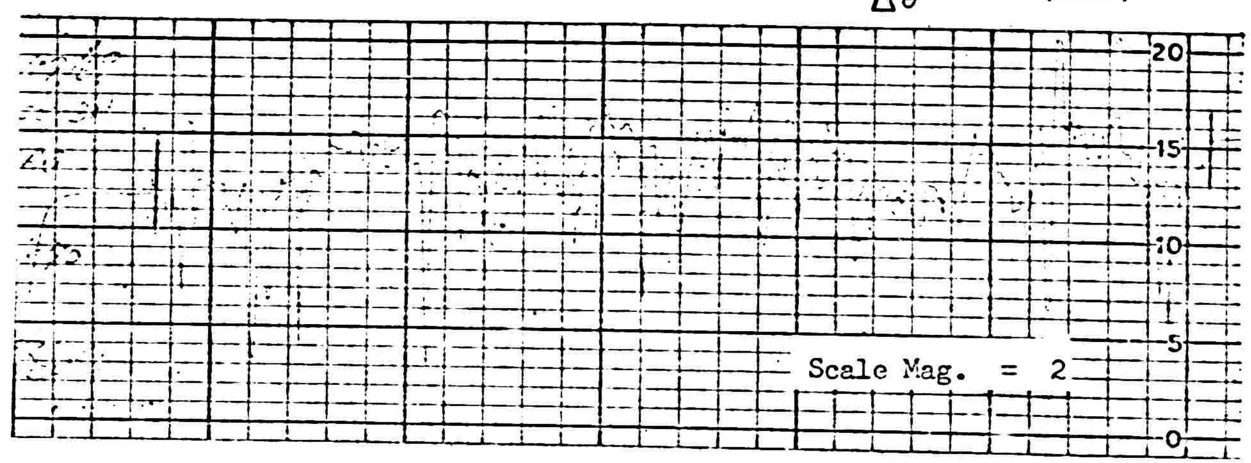


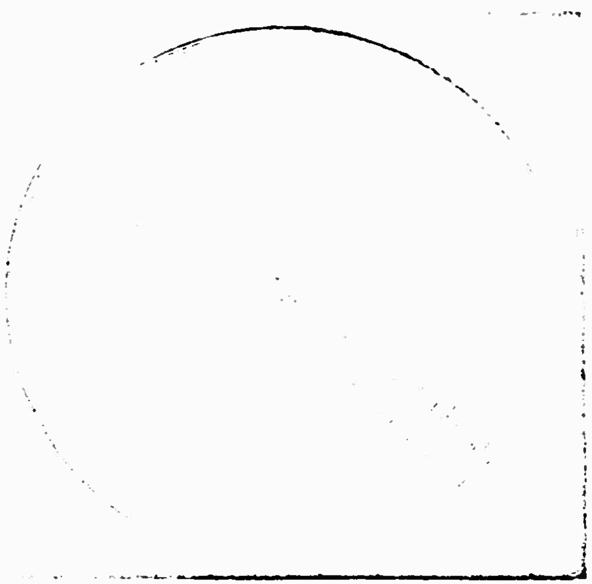
Photo Mag. = 4.5X

CLA = 70.0
RMS = 77.7

80 CS-3A (E) After Etch

θ MAX	31.0
θ MIN	(5.0)
$\Delta\theta$	(26.0)





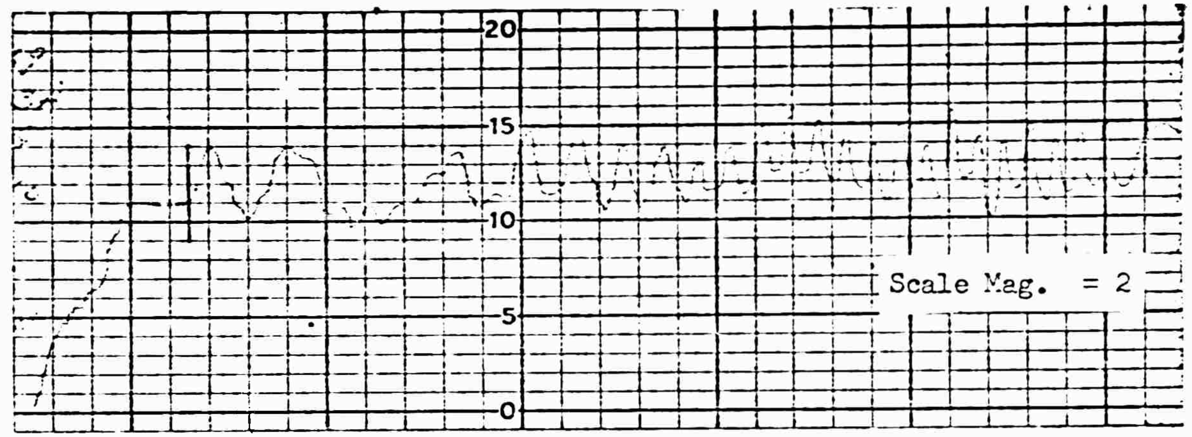


Photo Mag. = 3.7X

CLA = 60.0
RMS = 66.6

110 CS-3A (M) Before Etch

θ MAX	73.0
θ MIN	21.0
$\Delta\theta$	52.0

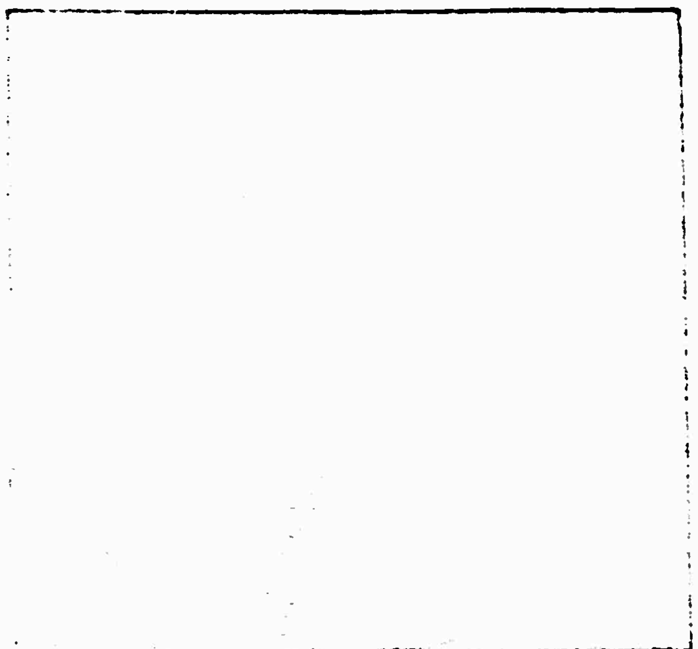
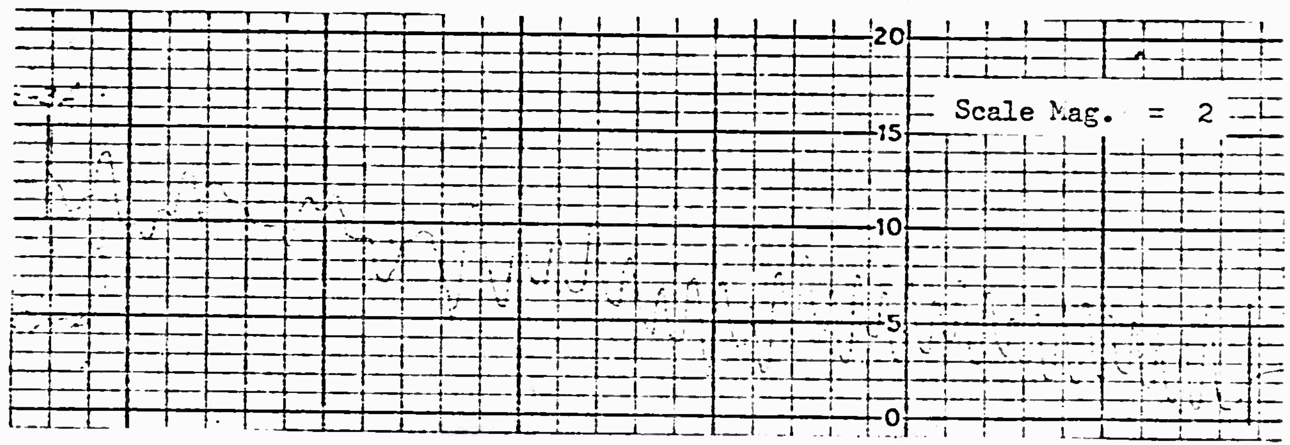


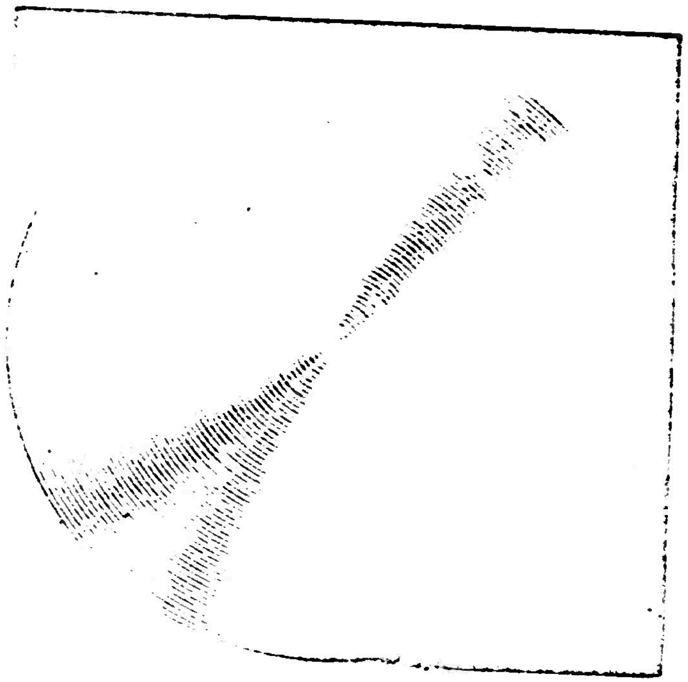
Photo Mag. = 4.5X

CLA = 70.0
RMS = 77.7

110 CS-3A (E) After Etch

θ MAX	32.0
θ MIN	(3.0)
$\Delta\theta$	(29.0





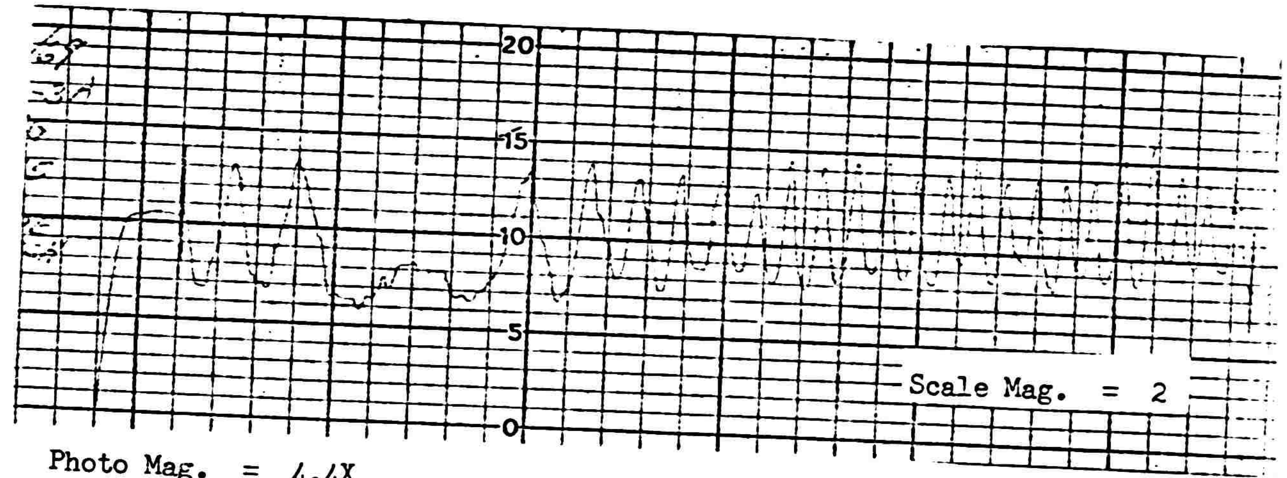


Photo Mag. = 4.4X

CLA = 95.0
RMS = 105.4

150 CS=3A (M) Before Etch

θ MAX	72.5
θ MIN	15.0
$\Delta\theta$	57.5

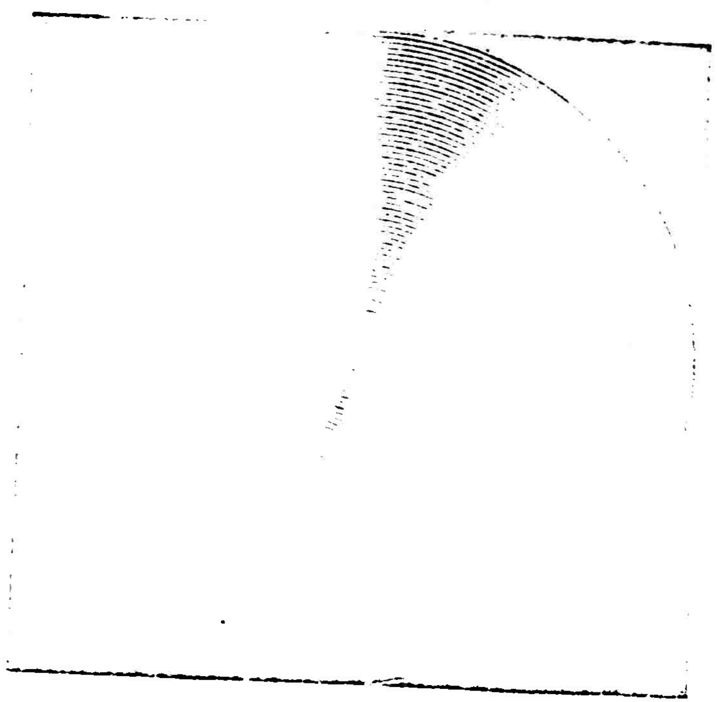
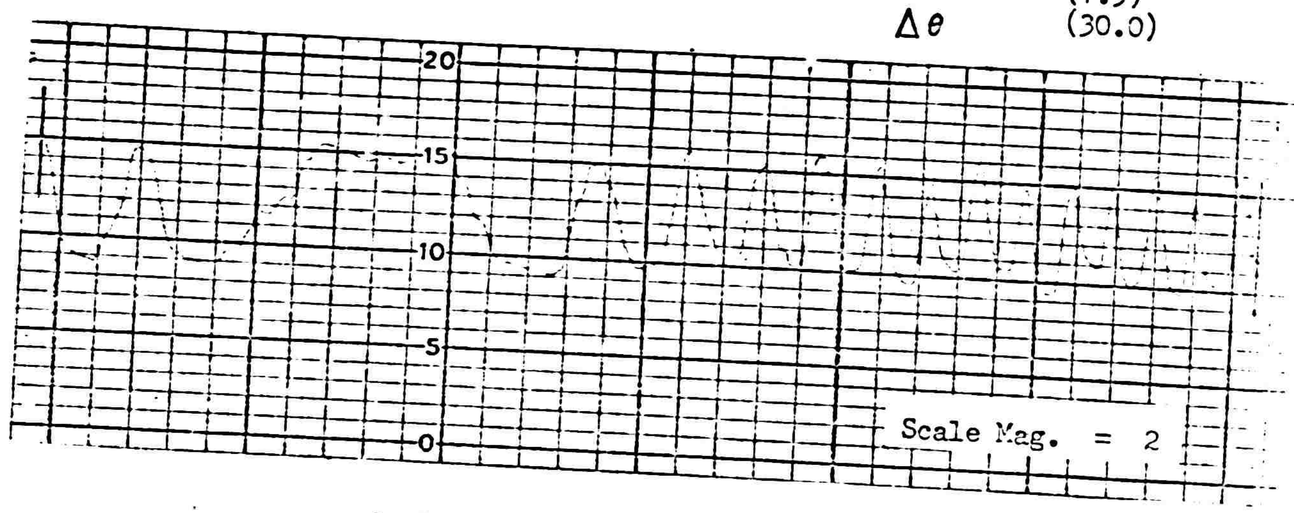


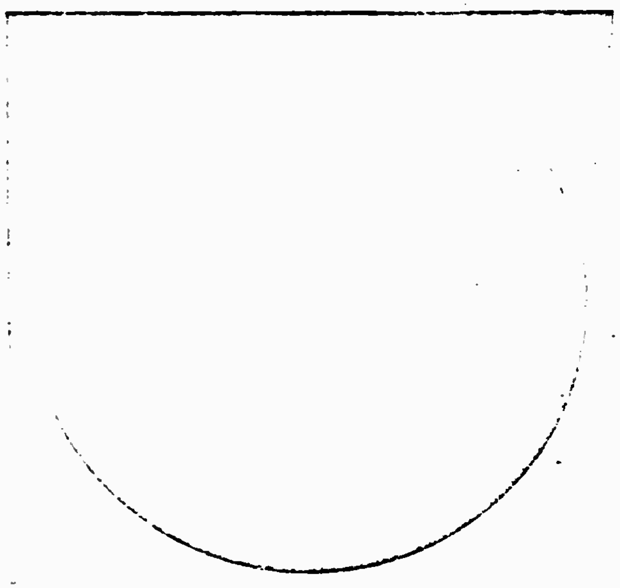
Photo Mag. = 4.5X

CLA = 70.0
RMS = 77.7

150 CS-3A (E) After Etch

θ MAX	37.5
θ MIN	(7.5)
$\Delta\theta$	(30.0)





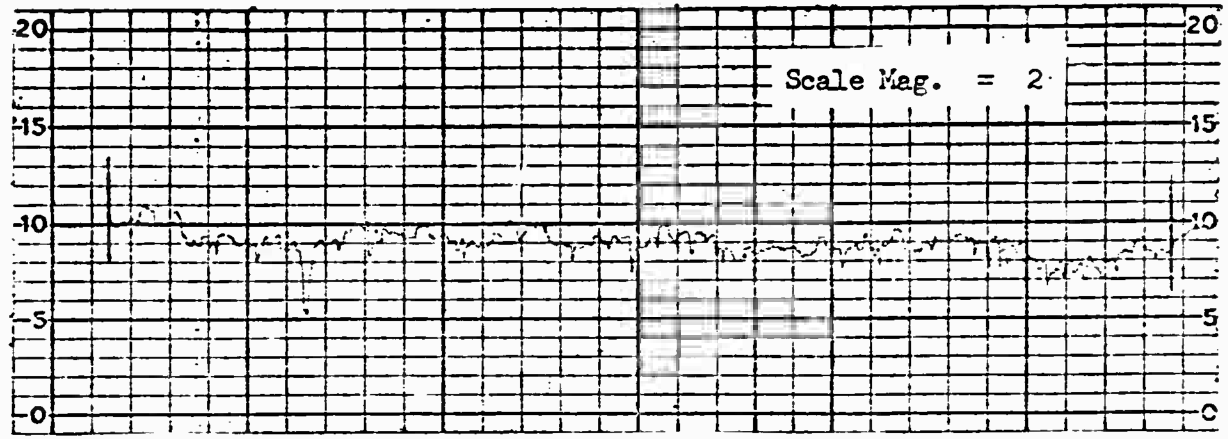


Photo Mag. = 3.7X

CLA = 22.0
RMS = 24.4

FGLP-CS-3A (M) Before Etch

θ MAX	52.0
θ MIN	11.0
$\Delta\theta$	41.0

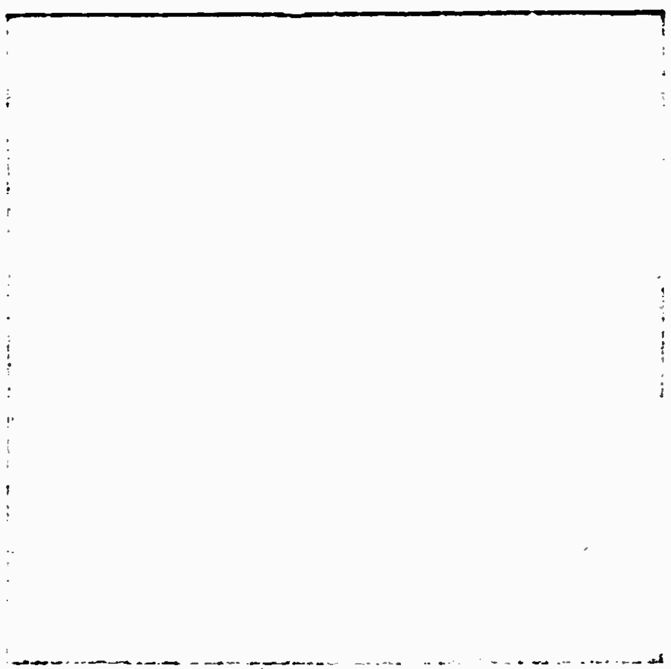
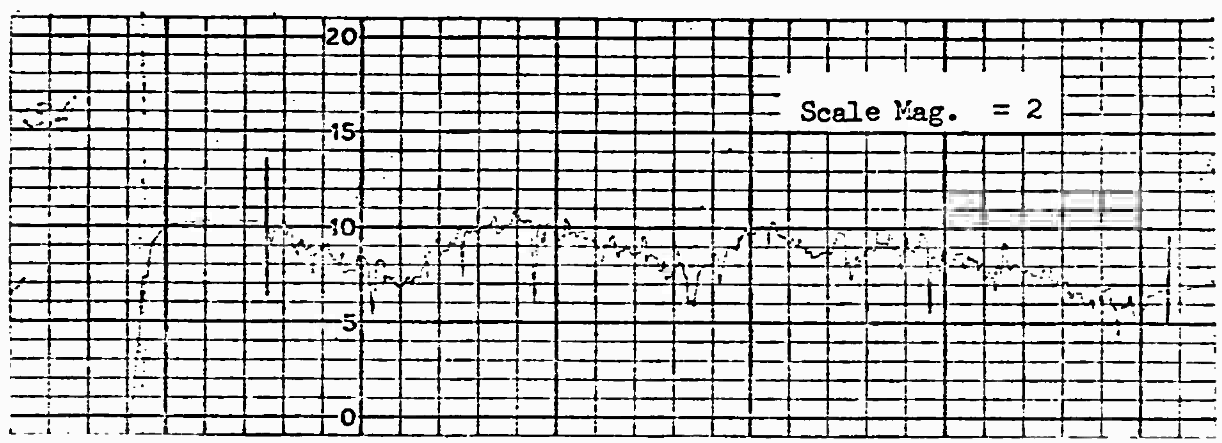


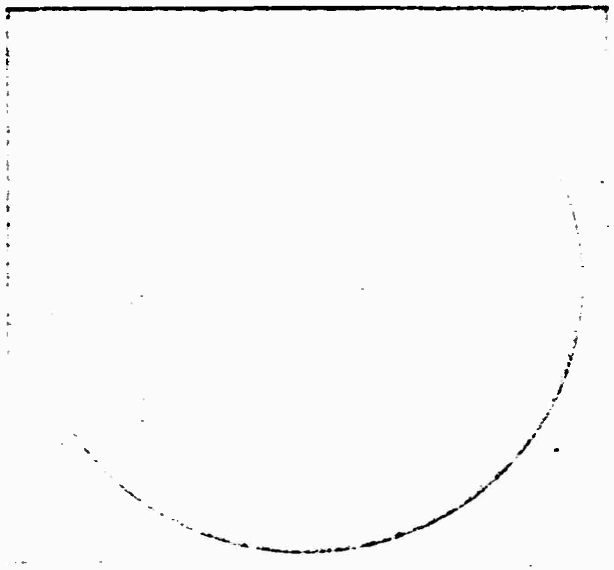
Photo Mag. = 4.5X

CLA = 41.0
RMS = 45.5

FGLP-CS-3A (E) After Etch

θ MAX	8.0
θ MIN	2.0
$\Delta\theta$	6.0





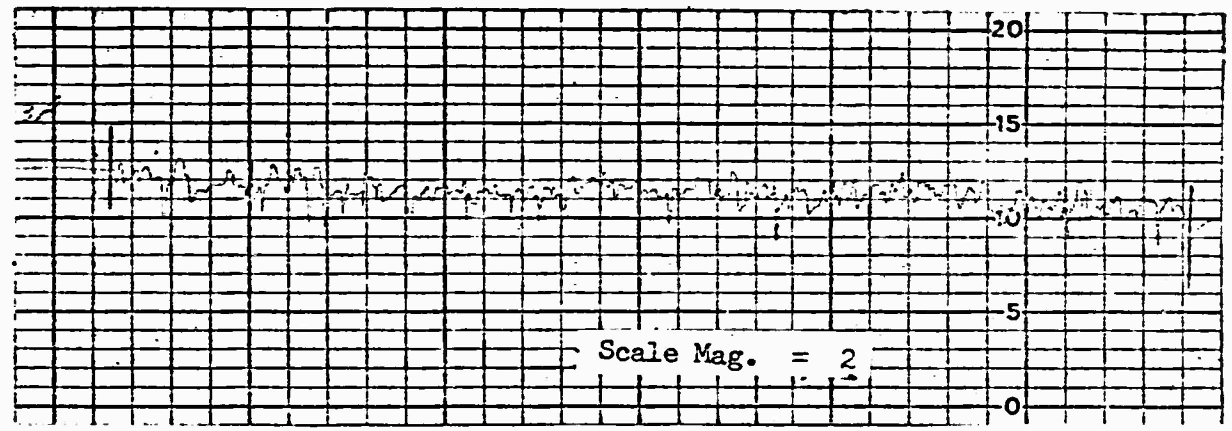


Photo Mag. = 37.X

CLA = 45.0
RMS = 50.0

FGHP-CS-3A (M) Before Etch

θ MAX	47.5
θ MIN	14.5
$\Delta\theta$	33.0

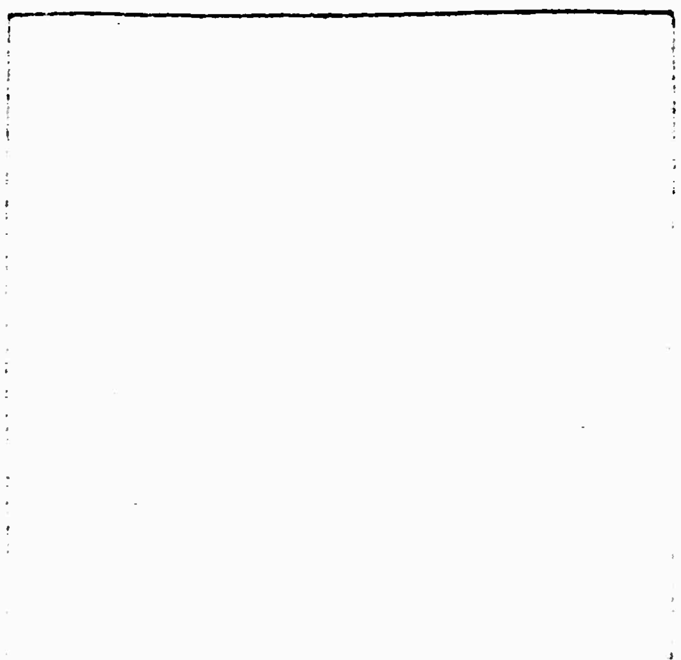
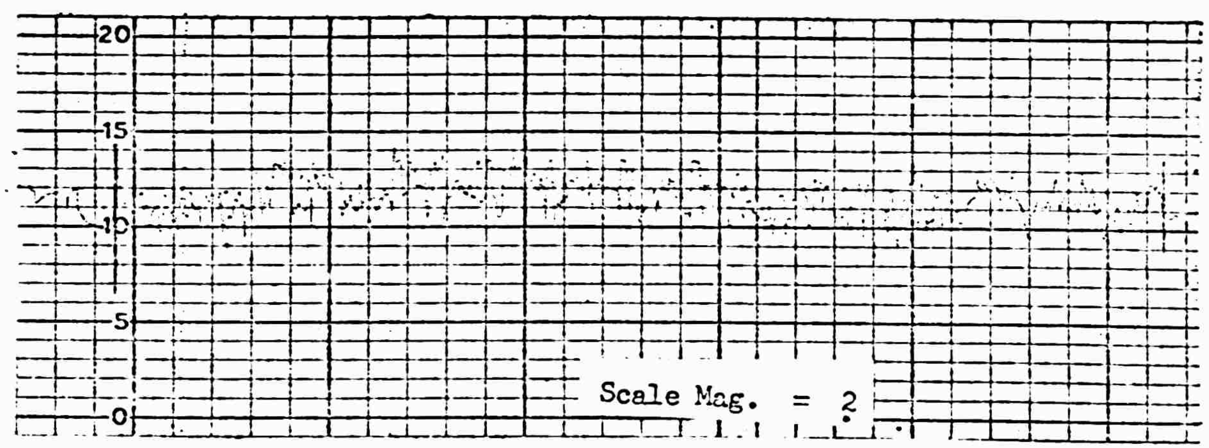


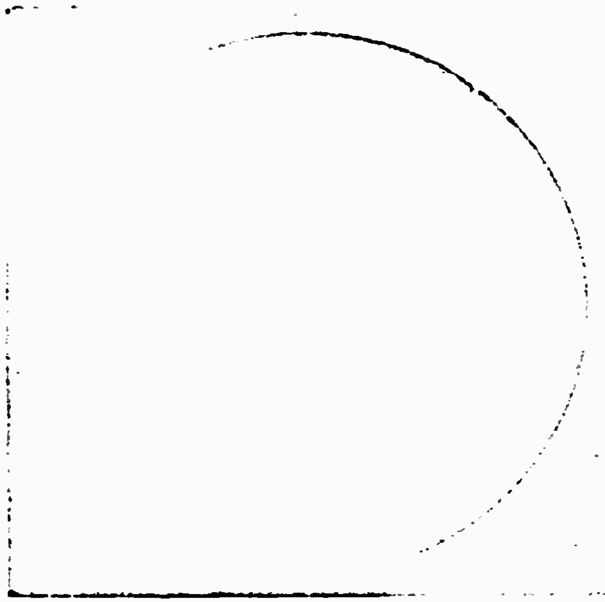
Photo Mag. = 4.5X

CLA = 48.0
RMS = 53.3

FGHP-CS-3A (E) After Etch

θ MAX	0.5
θ MIN	<0.5
$\Delta\theta$	—





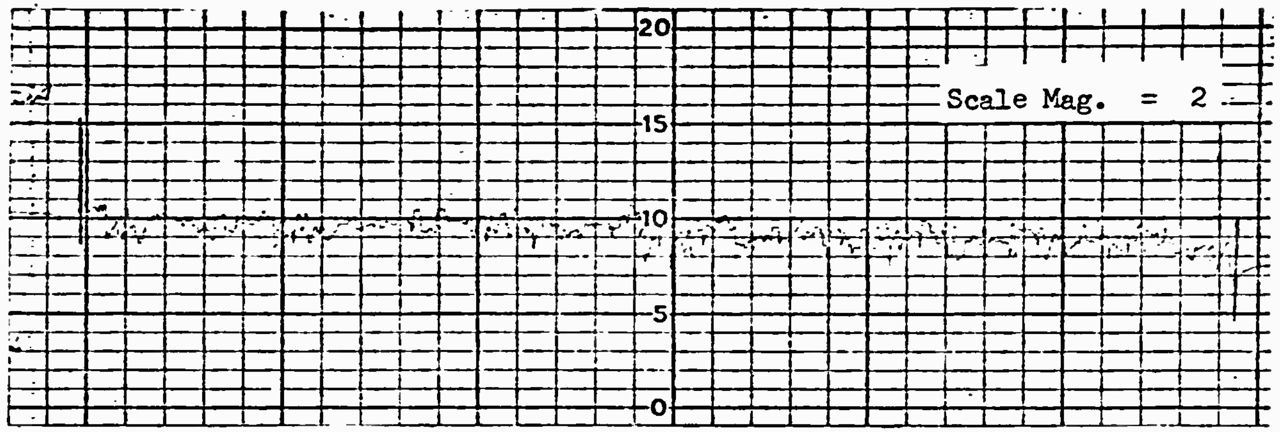


Photo Mag. = 3.7X

CLA = 25.0
RMS = 27.7

CGLP-CS-3A (M) Before Etch

θ MAX	61.0
θ MIN	17.0
$\Delta\theta$	44.0

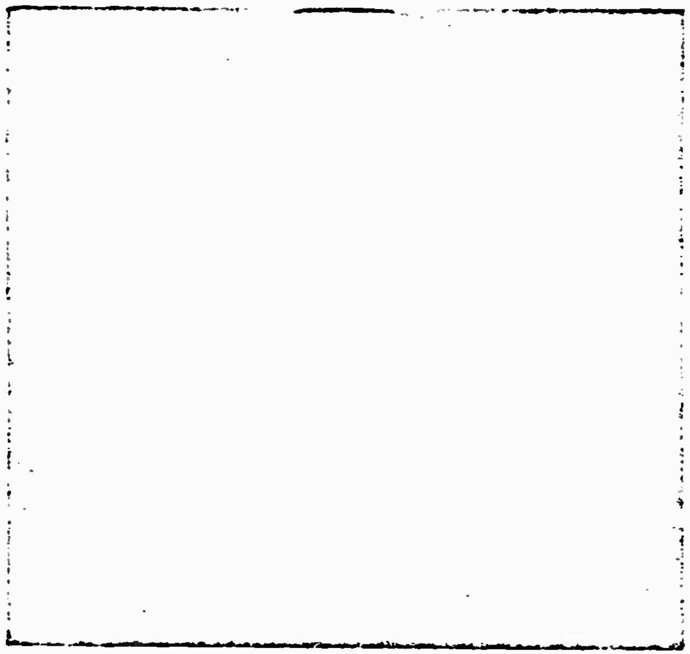
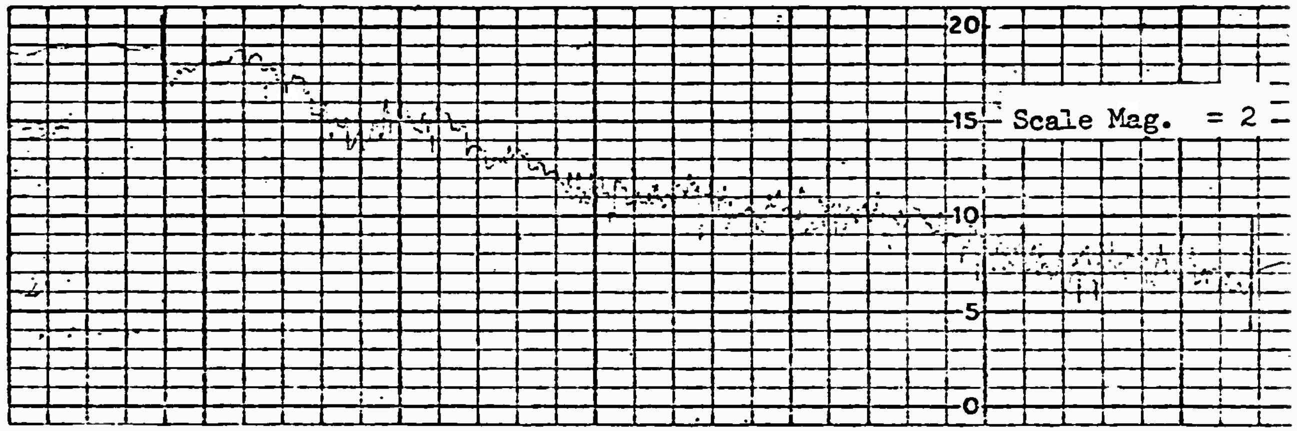


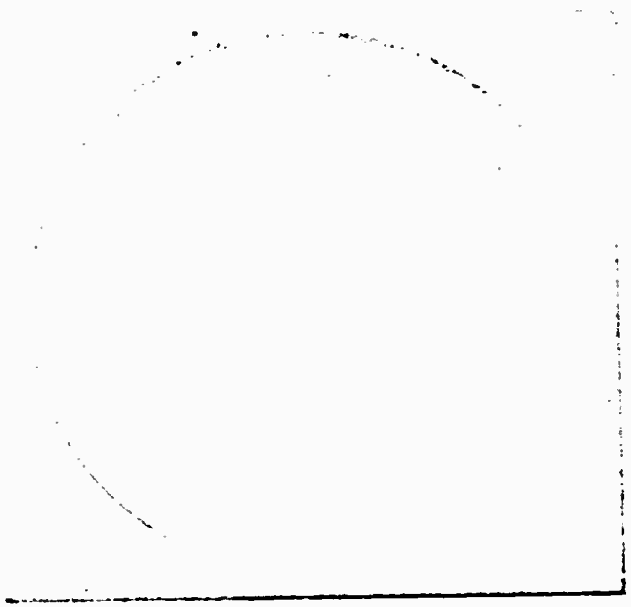
Photo Mag. = 4.5X

CLA = 35.0
RMS = 38.8

CGLP-CS-3A (E) After Etch

θ MAX	7.0
θ MIN	2.0
$\Delta\theta$	5.0





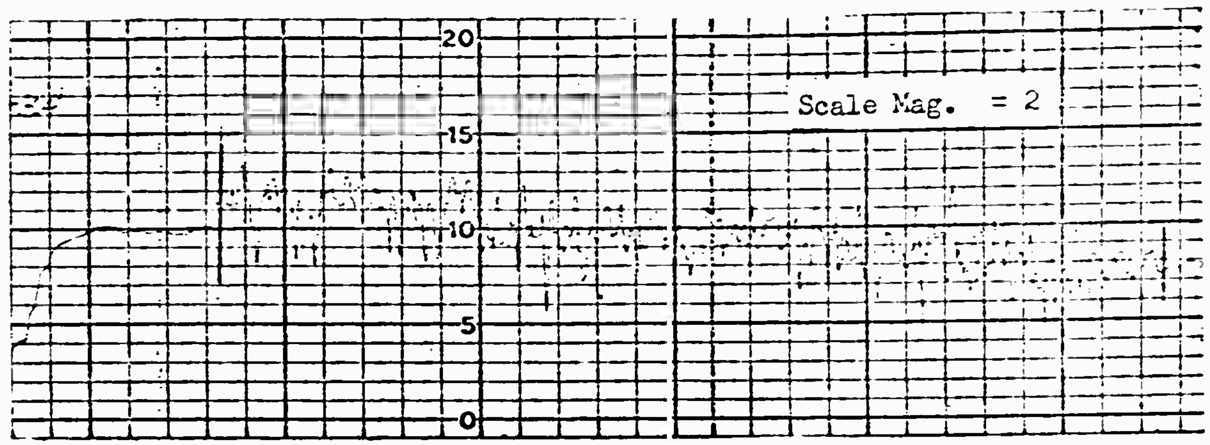


Photo Mag. = 3.7X

CLA = 48.0
RMS = 53.3

CGHP-CS-3A (M) Before Et.

θ MAX 76.0
θ MIN 4.0
$\Delta\theta$ 72.0

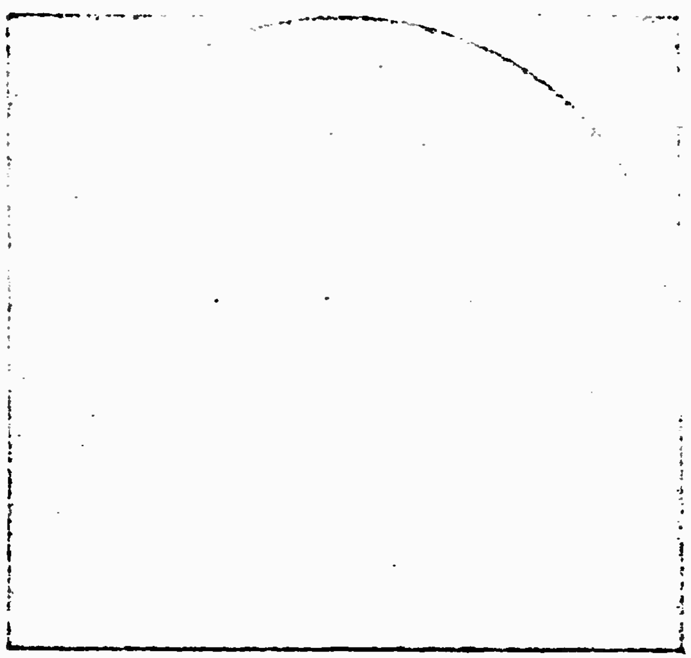
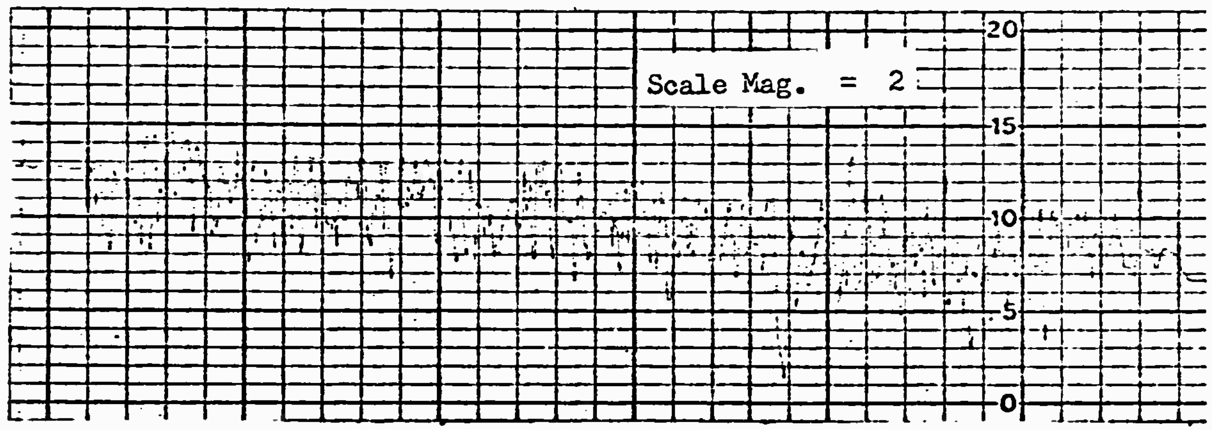


Photo Mag. = 4.5X

CLA = 80.0
RMS = 88.8

CGHP-CS-3A (E) After Etch

θ MAX 7.0
θ MIN 1.0
$\Delta\theta$ 6.0



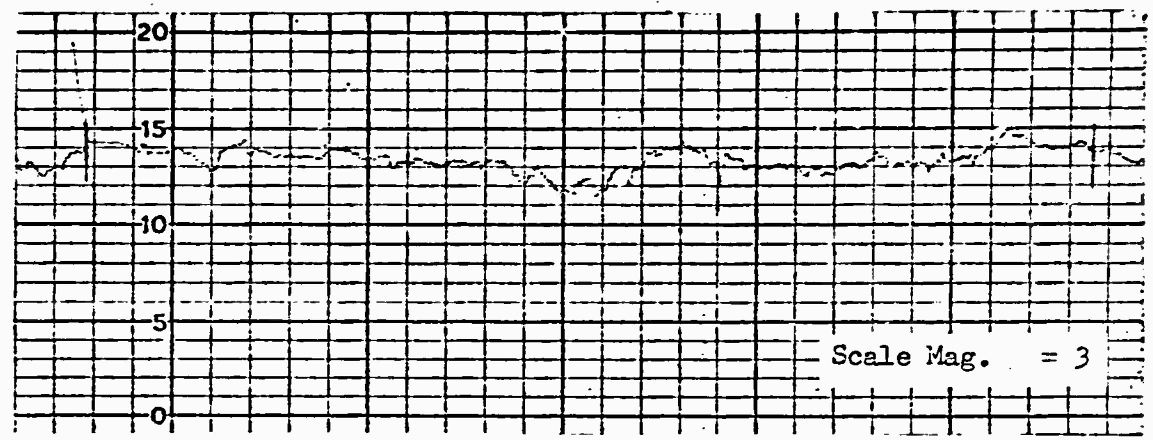


Photo Mag. = 2.7X

CLA = 1.2
RMS = 1.3

1 LS-3A (M) Before Etch

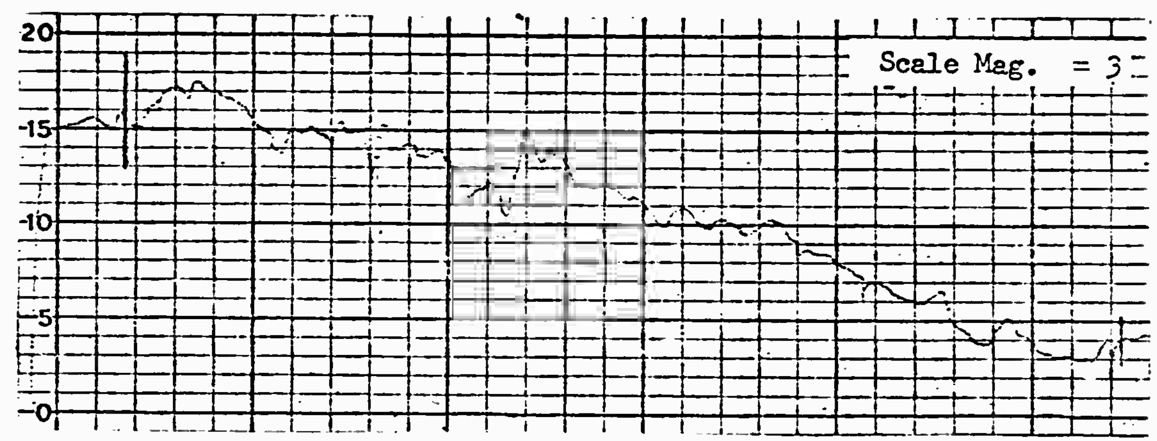
θ MAX 66.0
θ MIN 21.5
$\Delta\theta$ 44.5

Photo Mag. = 2.7X

CLA = 7.0
RMS = 7.8

1 LS-3A (E) After Etch

θ MAX 70.0
θ MIN (12.5)
$\Delta\theta$ (57.5)



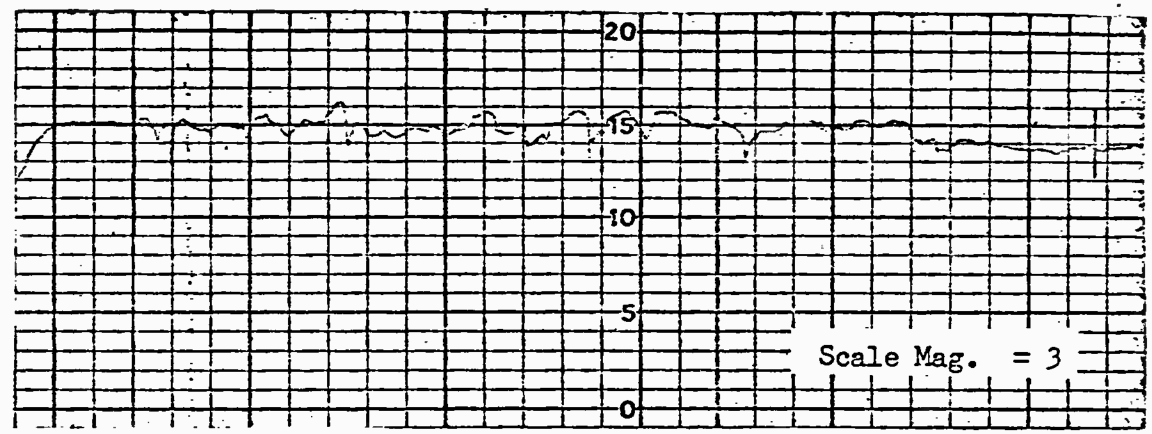


Photo Mag. = 2.7X

CLA = 4.5
RMS = 5.0

5 LS-3A (M) Before Etch

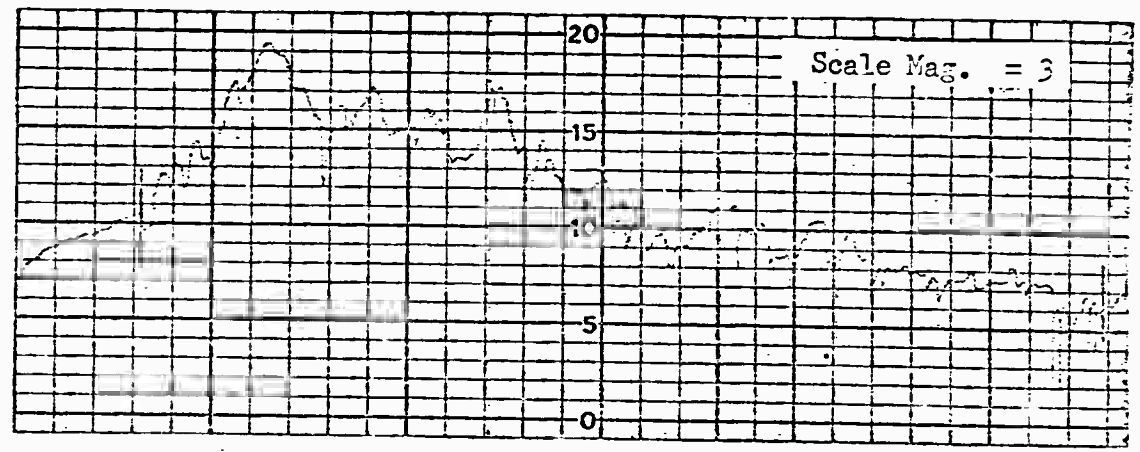
θ MAX 87.0
θ MIN 36.0
$\Delta\theta$ 51.0

Photo Mag. = 2.7X

CLA = 7.0
RMS = 7.8

5LS-3A (E) After Etch

θ MAX 74.0
θ MIN 19.0
$\Delta\theta$ 55.0



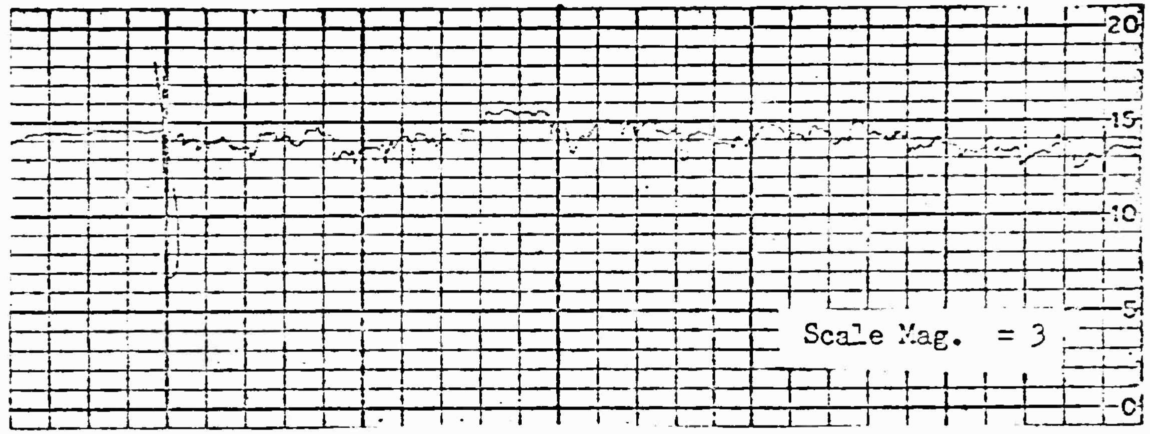


Photo Mag. = 2.9X

7 LS-3A (M) Before Etch

CLA = 7.2
RMS = 8.0

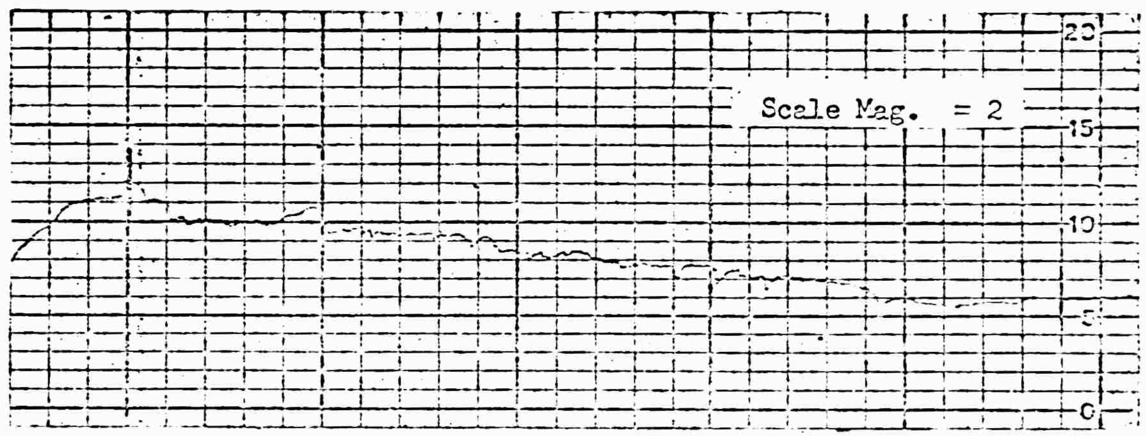
θ MAX 70.0
θ MIN 15.5
$\Delta\theta$ 54.5

Photo Mag. = 2.7X

7 LS-3A (E) After Etch

CLA = 9.0 - 9.5
RMS = 10.0 - 10.6

θ MAX 64.0
θ MIN (7.0)
$\Delta\theta$ (57.0)



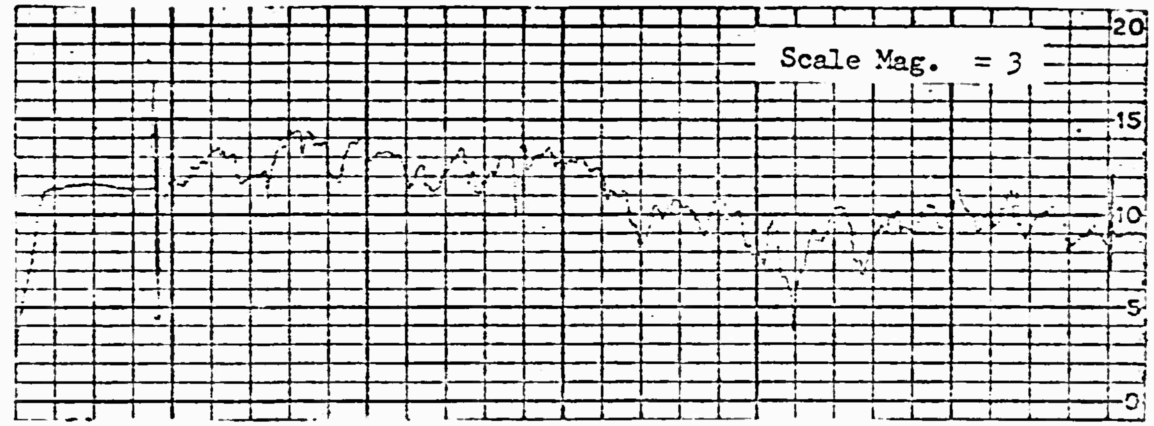


Photo Mag. = 2.7X

CLA = 8.5
RMS = 9.4

10 LS-3A (M) Before Etch

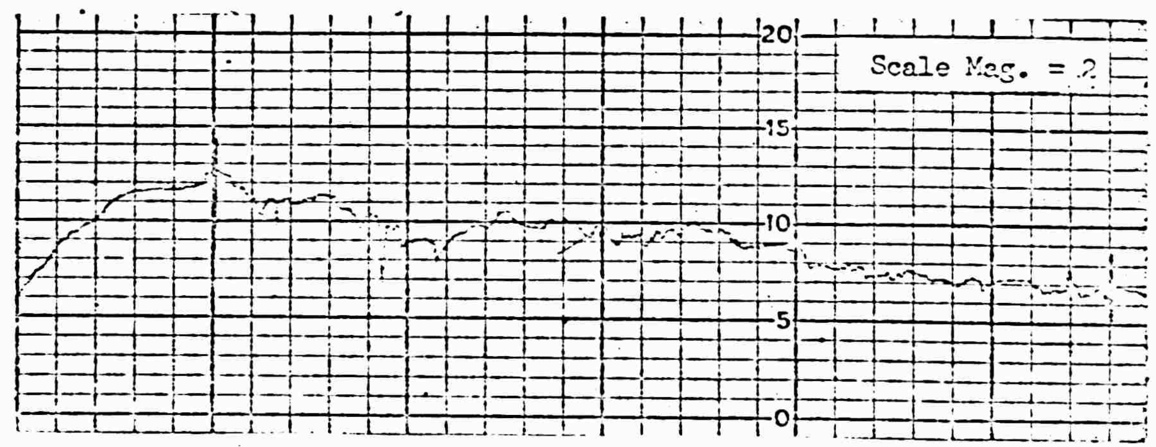
θ MAX	62.0
θ MIN	19.0
$\Delta\theta$	43.0

Photo Mag. = 2.7X

CLA = 10.8 - 11.0
RMS = 12.0 - 12.2

10 LS-3A (E) After Etch

θ MAX	79.0
θ MIN	10.5
$\Delta\theta$	68.5



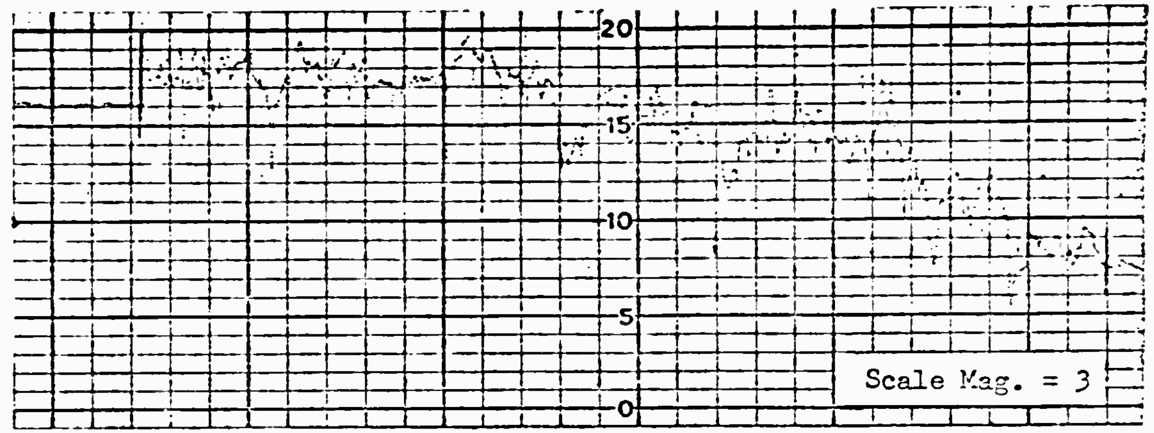


Photo Mag. = 2.7X

CLA = 17.0
RMS = 18.9

20 LS-3A (M) Before Etch

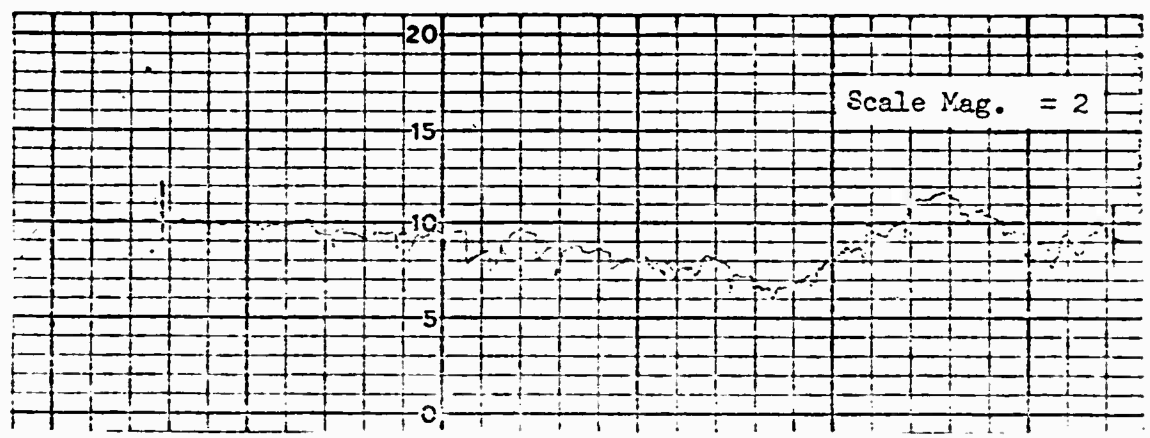
θ MAX	69.0
θ MIN	19.5
$\Delta\theta$	49.5

Photo Mag. = 2.7X

CLA = 32.0
RMS = 35.5

20 LS-3A (E) After Etch

θ MAX	62.0
θ MIN	16.0
$\Delta\theta$	46.0



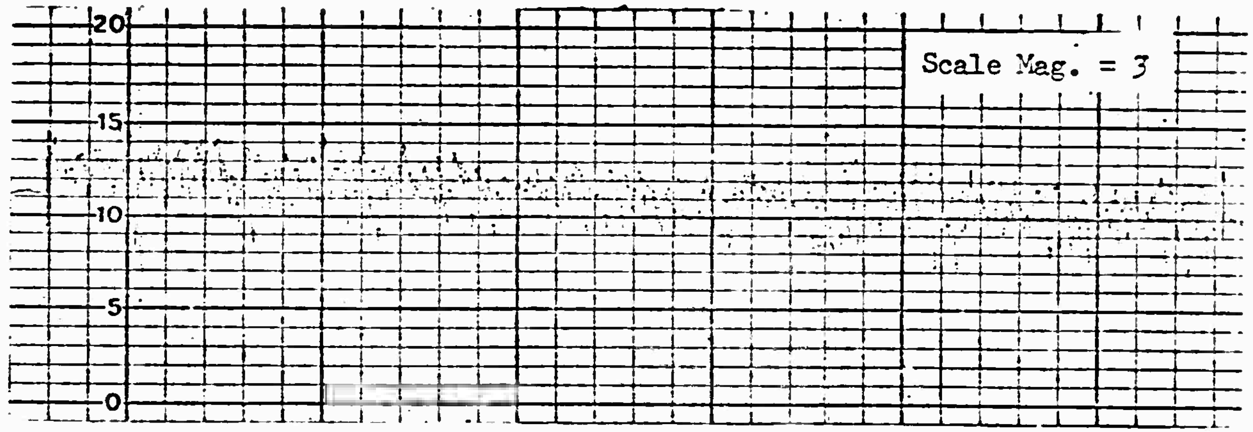


Photo Mag. = 2.9X

CLA = 21.0
RMS = 23.3

40 LS-3A (M) Before Etch

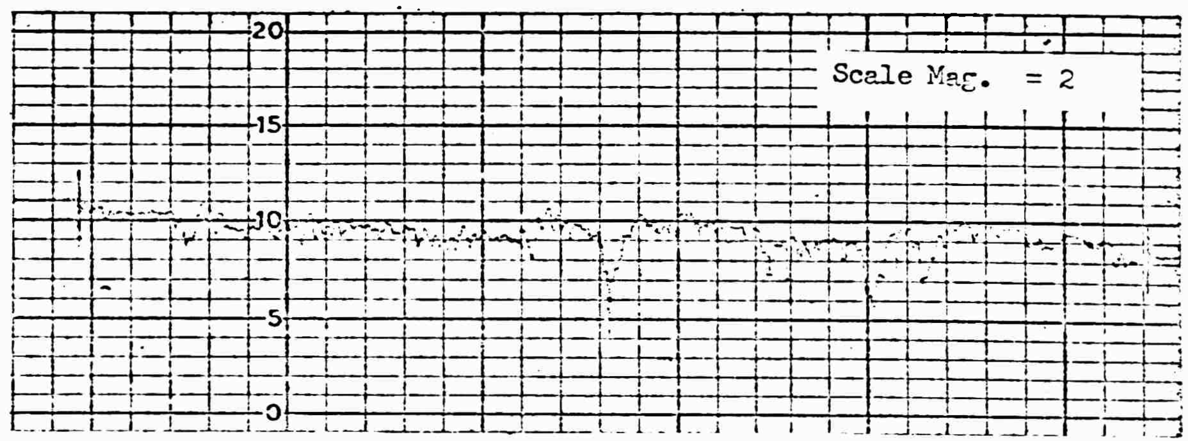
θ MAX	75.0
θ MIN	14.0
$\Delta\theta$	61.0

Photo Mag. = 2.9X

CLA = 22.0
RMS = 24.4

40 LS-3A (E) After Etch

θ MAX	23.5
θ MIN	7.5 (irregular droplet)
$\Delta\theta$	16.0



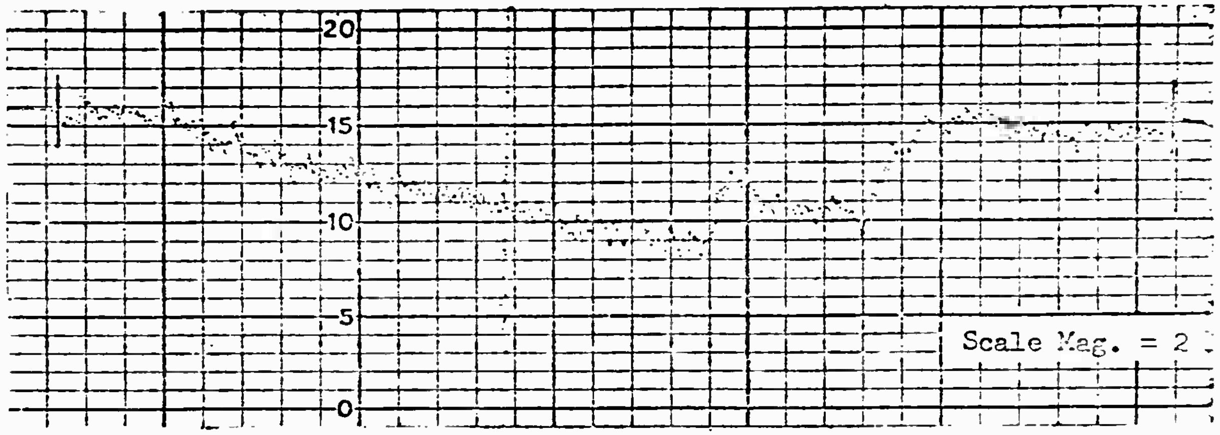


Photo Mag. = 2.9X

CLA = 36.0 - 38.0
RMS = 40.0 - 42.0

80 LS-3A (M) Before Etch

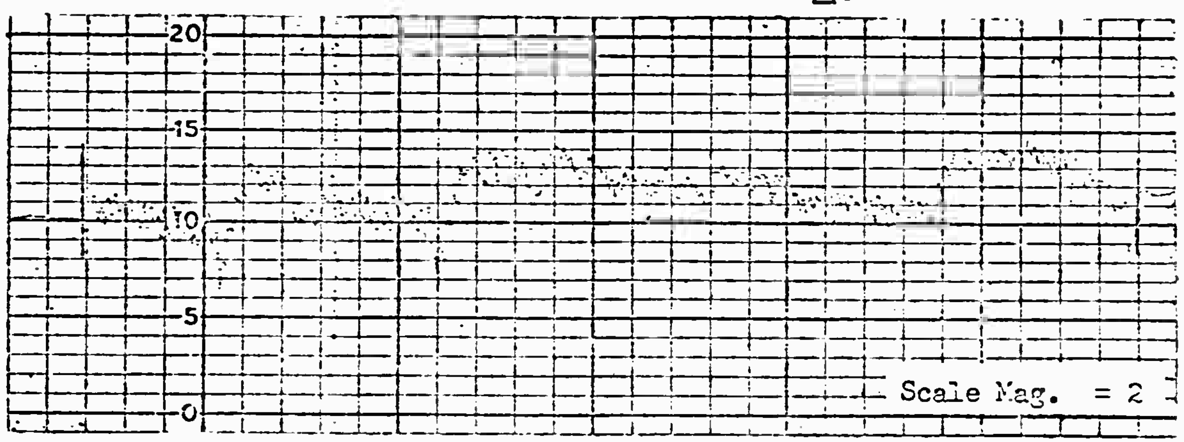
θ MAX 63.0
θ MIN 25.0
$\Delta\theta$ 38.0

Photo Mag. = 2.8X

CLA = 32.0
RMS = 35.5

80 LS-3A (E) After Etch

θ MAX 56.0
θ MIN 13.5
$\Delta\theta$ 42.5



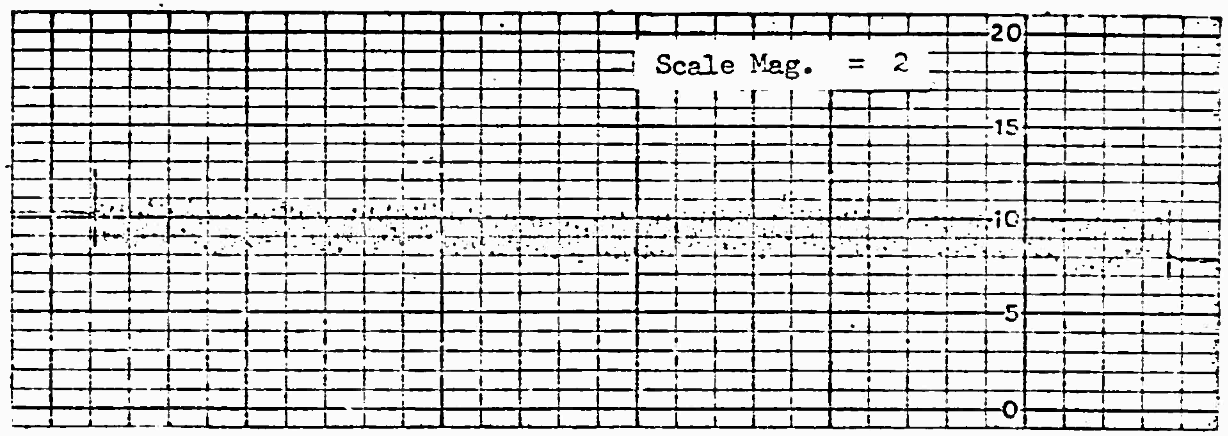


Photo Mag. = 2.9X

CLA = 47.0
RMS = 52.2

110 LS-3A (M) Before Etch

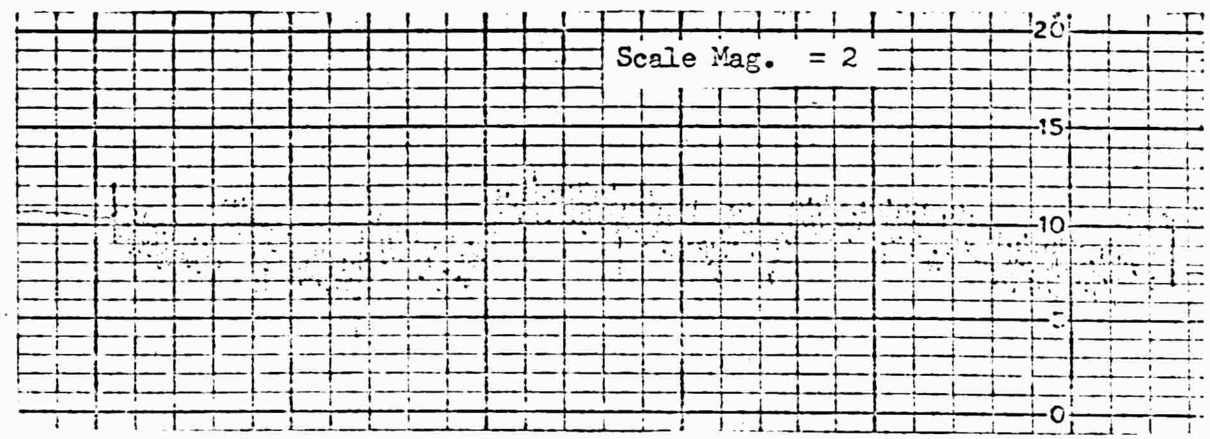
θ MAX 66.5
θ MIN 26.5
$\Delta\theta$ 40.0

Photo Mag. = 2.8X

CLA = 58.0
RMS = 64.4

110 LS-3A (E) After Etch

θ MAX 17.0
θ MIN 2.5 (Irregular droplet)
$\Delta\theta$ 14.5



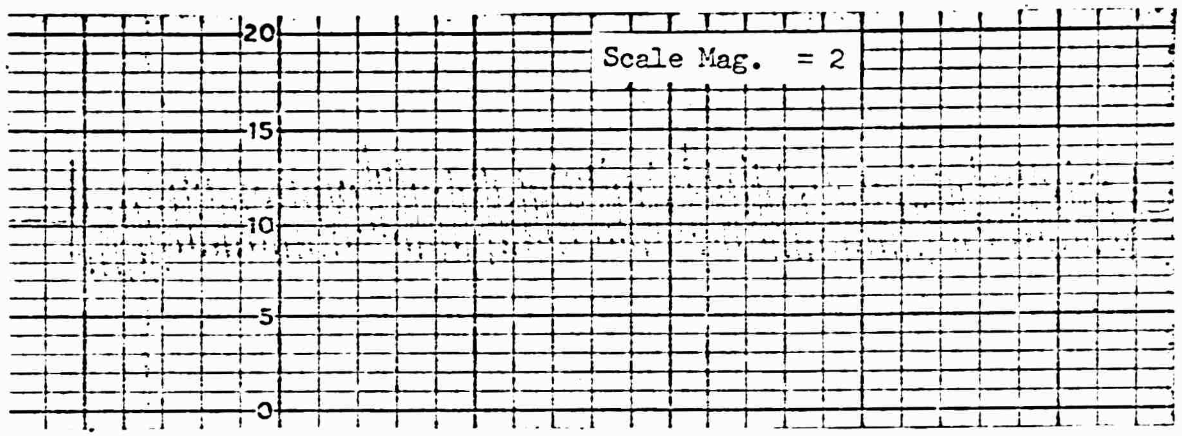


Photo Mag. = 2.9X

CLA = 72.0
RMS = 80.0

150 LS-3A (M) Before Etch

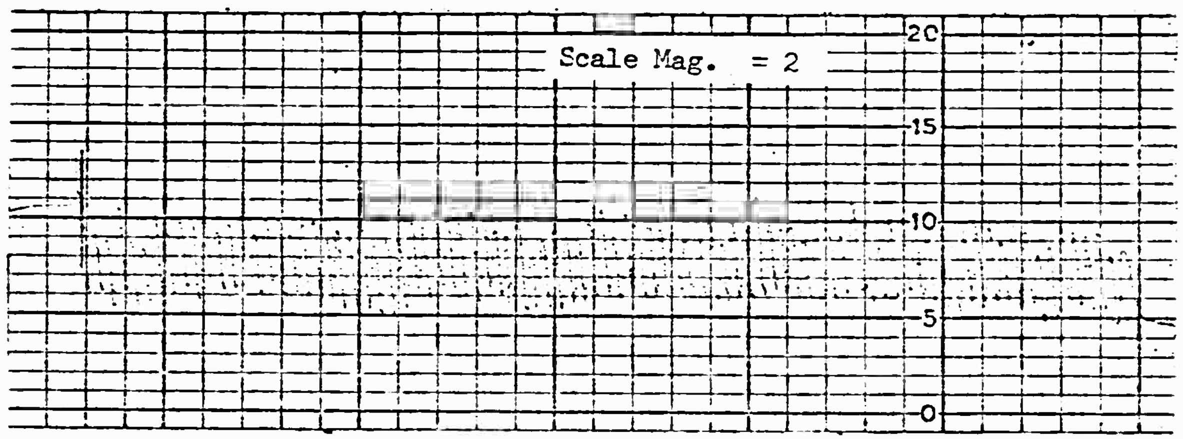
θ MAX 79.0
θ MIN 22.0
$\Delta\theta$ 57.0

Photo Mag. = 2.8X

CLA = 72.0
RMS = 80.0

150 LS-3A (E) After Etch

θ MAX 86.0
θ MIN 19.0
$\Delta\theta$ 67.0



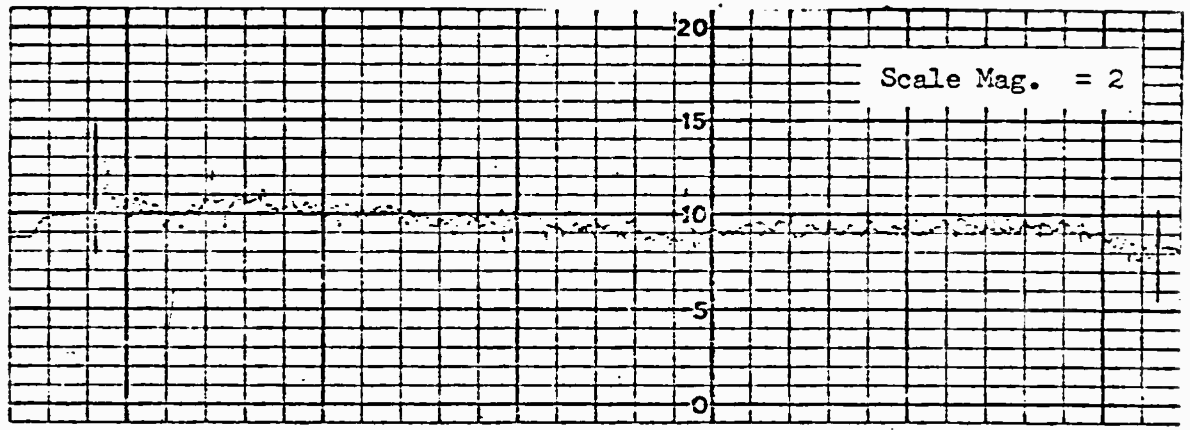


Photo Mag. = 2.7X

CLA = 20.0
RMS = 22.2

FGLP-LS-3A (M) Before Etch

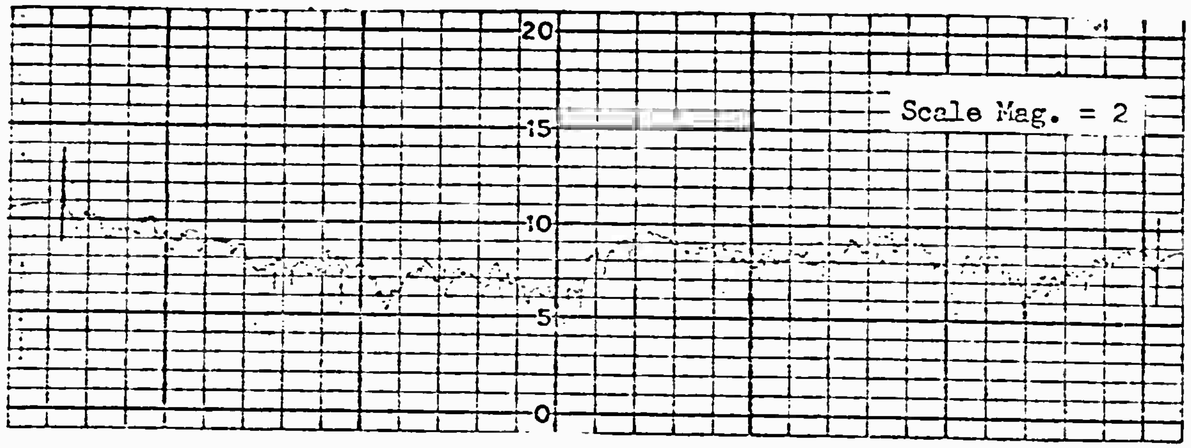
θ MAX 58.5
θ MIN 10.5
$\Delta\theta$ 48.0

Photo Mag. = 2.8X

CLA = 20.0
RMS = 22.2

FGLP-LS-3A (E) After Etch

θ MAX 74.0
θ MIN 18.5
$\Delta\theta$ 55.5



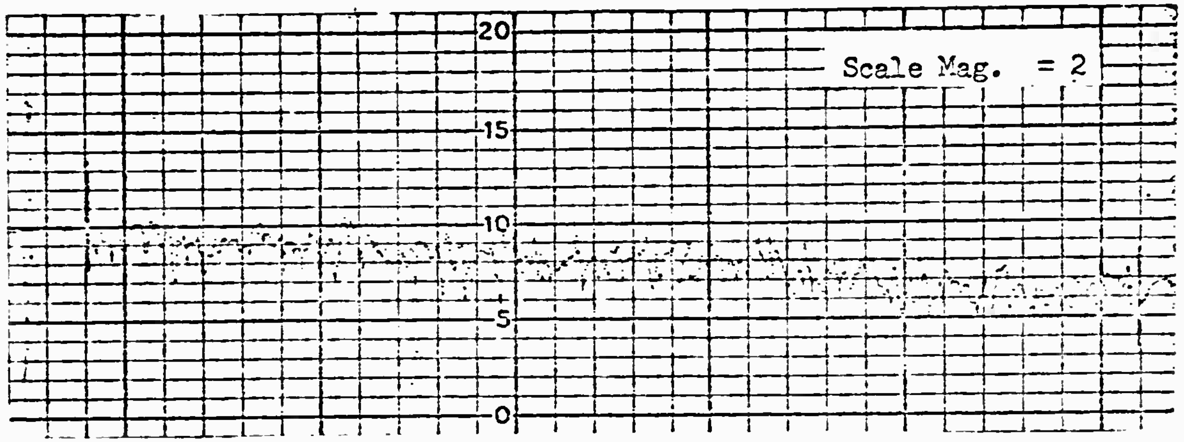


Photo Mag. = 2.7X

CLA = 32.5
RMS = 35.6

FGHP-LS-3A (M) Before Etch

θ MAX 39.0
θ MIN 14.0
$\Delta\theta$ 25.0

Photo Mag. = 2.8X

CLA = 45.0
RMS = 50.0

FGHP-LS-3A (E) After Etch

θ MAX = 63.5
θ MIN = 15.0
$\Delta\theta$ 48.5

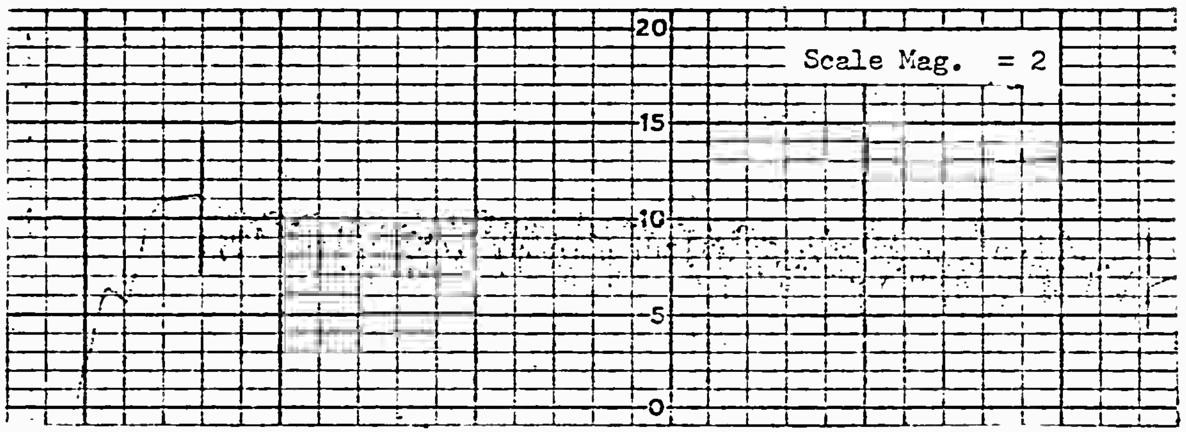


Photo Mag. = 2.7X

CLA = 34.0
RMS = 38.8

CGLP-LS-3A (M) Before Etch

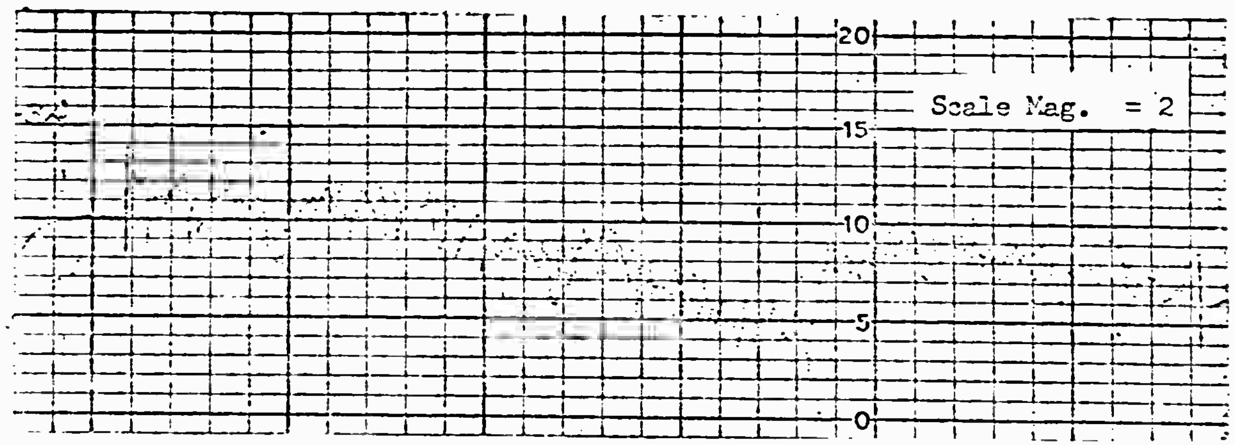
θ MAX	81.5
θ MIN	18.0
$\Delta\theta$	63.5

Photo Mag. = 2.8X

CLA = 36.0
RMS = 39.9

CGLP-LS-3A (E) After Etch

θ MAX	13.0
θ MIN	3.0
$\Delta\theta$	10.0



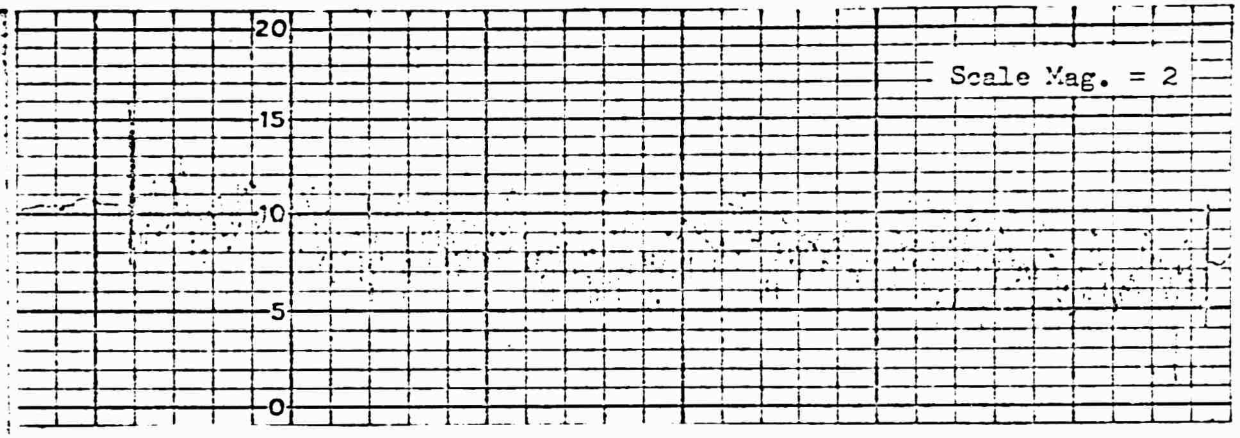


Photo Mag. = 2.7X

CLA = 55.0
RMS = 61.0

CGHP-LS-3A (M) Before Etch

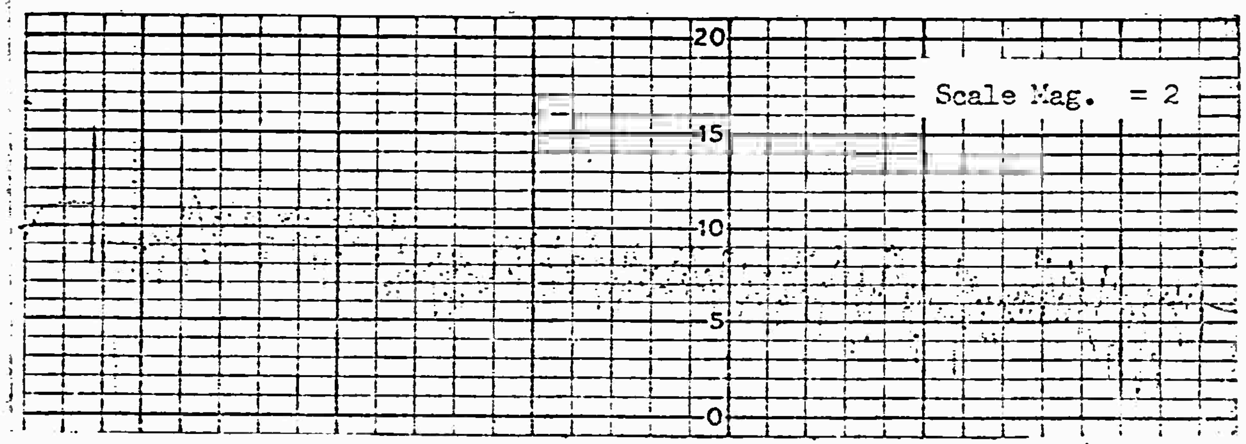
θ MAX	83.5
θ MIN	25.0
$\Delta\theta$	58.5

Photo Mag. = 2.8X

CLA = 60.0
RMS = 66.6

CGHP-LS-3A (E) After Etch

θ MAX	88.0+
θ MIN	31.0
$\Delta\theta$	57.0



2. Preparation for Bonding

The schedule for bond preparation, etched surface evaluation, and adhesive bonding was carefully arranged to benefit both data-taking and bonding. Three hours after etching had been completed, the curing agent was added to the adhesive resin mixture, with the bonds being completed within 30 minutes from the start of that mixing. Twelve specimens were prepared each day for two days, with fifteen specimens being prepared the third day, for a given type of specimen. Three such series covered the butt tensile, core shear, and lap shear specimen types.

The substrate cleaning and etching was conducted according to Avco RAD Specification G100004-4, "Immersion Cleaning and Etching of Aluminum and Aluminum Alloy, Process for" except that manual solvent wiping with methyl-ethyl ketone (MEK) replaced the recommended vapor degreasing. Distilled water was used for the final rinse.

3. Adhesive Bonding

The adhesive formula for each of nine batches was:

Epoxy resin	Epon 828	50.00 grams
Viscosity adjuster	Cab-o-sil	1.25 grams
Total prepared resin		51.25 grams
Hardener	DETA	5.00 grams
Total mixture		56.25 grams

The epoxy resin and fumed silica were mixed together in two master batches, each consisting of 750.00 grams of resin and 18.75 grams of Cab-o-sil. Prior to mixing, the Cab-o-sil was dried for four hours at 230°F. After mixing the batches were placed under full vacuum for at least 10 minutes at room temperature to remove air and volatiles. The master batches were stored at room temperature. A day's batch of prepared resin and hardener was mixed by hand for two minutes and then spread thin to maximize evolution of reaction exotherm heat. This procedure minimized mixture aging over the 30-minute application period. Curing in all cases was conducted at room temperature (72° ±4) for a minimum of 24 hours in order to avoid thermal expansion variability.

The cylindrical substrates were aligned and cured in Vee-blocks using substrate length measurements to set the 0.006 inch bond-line thickness. The lap-shear specimens, however, required a special set-up procedure. Because differing surface finishes were machined, lapped, fly-cut on the specimen surfaces, more or less material was removed from some specimens, yielding specimens which varied in the thickness by 3-4 mils from the original blanking stock.

All specimens were dry fitted, shimming individual specimens with plastic (mylar) film such that a 1 mil feeler guage blade could not be inserted between the mating halves at the point to be bonded.

Previous experiments with the bonding system (adhesive) used in this program permitted bonds of approximately 6 mil thickness to be obtained on lap shear specimens when a "dead weight" of 100 grams per sq. in. was applied to the buttered halves. The weight-adhesive viscosity combination allowed the substrates to seek that equilibrium which, in previous experiments, yielded an approximately 6 mil thick bond line. Individual lap shear substrates were pre fitted in a bonding fixture specifically designed to maintain the alignment and overlap required. Bonding fixtures were made to bond six individual lap shear specimens simultaneously. One substrate of each of six specimens was placed onto the fixture and spring loaded. The second or mating substrate was placed on the fixture and a straight edge was placed across the 0.50 in. overlap area of all six specimens. Mlyar and/or polyethylene film shim stock was added as necessary under the lower individual substrates so that less than one mil clearance was noted between the suspended straight edge and any singular substrate (in the overlap area). It should be noted that the fixtures were initially designed for mating 0.064 in. nominal thickness substrates yielding an approximate 5 mil parallel bond line. Following surface preparation the adhesive was prepared and spread into a thin film. Each substrate was wet with adhesive, covering the sample width and approximately 0.625 in. of the bonding edge. Samples were permitted to set at room temperature about 20 minutes to permit application of adhesive to all samples, allow excess air to escape and ensure a more uniform consistency viscosity-wise (adhesive). Specimens were then assembled and a 100 gram per square inch (overlap area) load was applied by means of a pre weighed bar. Room temperature cure of 24 hours was effected before specimens were removed from the fixtures.

4. Nondestructive Test Evaluation

a. Radiography

A few butt tensile specimens were radiographed at an oblique angle. A sensitivity of 2-1T with an aluminum penetrameter (0.015" thk, 0.008" dia. hole) was obtained. A polyethylene film step wedge (0.005" and 0.010" thick) was constructed and placed across the bond area. The polyethylene film could not be adequately discriminated in the radiographs. Similar lack of ability to radiographically observe bubbles in the bond line was experienced with the lap shear and core shear specimens.

Radiographic conditions:

Using a Philips-Norelco 50-150 KV X-ray tube with a 2.5 mm focal spot operated at 100 KV, 10 Ma for 1 minute and a focus to film distance of 48 inches, representative radiographs were obtained on Eastman Kodak type "M" film. Films were hand processed (developed) for 8 minutes at 68°F. The tube was positioned at an angle of 30° off the perpendicular such that the area of interest was projected onto the film for the butt tensile specimens.

b. Ultrasonic Pulse-echo

The centering holes and load-pin holes in the butt tensile specimens made it impossible to obtain unambiguous bond/unbond indications.

The lap shear specimens were all inspected for bond/unbond, and no unbonds were detected. Reference bond/unbond specimens were prepared from two pairs of substrates using coupling agent to represent the bonded case.

Equipment used:

Branson Sonoray, Model 51C

A.I. 5.0 MHz/.312 dia. type SFZ transducer

Delay 5	Damping $9\frac{1}{2}$
Range 6	C Gain A
Reject 1 o'clock	Extended Range 3 o'clock

The core shear specimens were all inspected for bond/unbond, and no unbonds were detected. Reference bond/unbond specimens were prepared from two pairs of substrates using coupling agent for the bonded case.

Equipment used:

Branson Sonoray, Model 51C

AI 5.0 MHz/.312 dia. SFZ transducer

Delay 5	Damping 10
Range $6\frac{1}{2}$	C Gain A
Reject 1 o'clock	Extended Range 3 o'clock

Measured bond-line thicknesses are presented in Table III for comparison.

5. Destructive Test Evaluation

Results of destructive tests are presented in Table IV. The specimens were all pulled at room temperature, 75° ±2°F and 50% RH, on the Instron universal testing machine. Crosshead speed was adjusted to provide an applied stress rate of 600 to 700 psi per minute.

6. Correlation and Analysis

No correlation and analysis studies have been conducted to date. The entire content of data and experience with these specimens will be sifted via correlations to extract the meaningful relationships.

TABLE III

BOND LINE THICKNESS

mils

Surface Finish Nominal	BUTT TENSILE 1A	2A	3A	CORE SHEAR 1A	2A	3A	LAP SHEAR 1A	2A	3A
1 RMS	5.5	3.8	4.9	5.7	4.8	4.2	2.1	3.9	2.7
5 RMS	4.4	4.2	4.4	5.5	6.3	4.6	2.6	1.0	3.2
7 RMS	10.0	4.7	6.7	5.2	4.9	3.9	5.0	2.0	1.3+
10 RMS	7.1	6.6	4.2	5.1	3.6	5.0	1.2	2.3	3.5
20 RMS	7.6	6.5	5.5	6.3	9.7	7.4	3.9	3.1	1.9
40 RMS	6.1	6.5	5.2	8.8	8.2	7.9	4.5	4.6	3.6
80 RMS	4.7	6.4	6.7	7.4	5.5	5.2	7.2	6.1	4.4
110 RMS	8.3	7.5	8.1	5.6	5.2	8.2	3.6	5.9	6.4
150 RMS	7.7	4.9	6.0	6.0	7.5	6.4	5.0	2.7	2.8
FGLP	6.1	5.6	4.5	8.1	6.6	5.8	3.7	4.4	3.9
FGHP	6.6	5.8	5.5	5.5	4.6	4.9	3.7	3.0	4.8
CGLP	7.3	6.0	4.6	6.0	7.3	5.7	2.2	1.3	2.7
CGHP	4.4	2.7	5.3	3.9	5.4	4.0	3.0	5.2	5.3

TABLE IV

ADHESIVE BOND ULTIMATE STRENGTH
pounds per square inch

Surface Finish Nominal	BUTT TENSILE			CORE SHEAR			LAP SHEAR		
	1A	2A	3A	1A	2A	3A	1A	2A	3A
1 RMS	3900	3750	3200	3140	4500	460	1360	1320	910
5 RMS	5770	4780	5540	1280	1470	2870	1260	1390	910
7 RMS	3600	3920	5190	1610	1590	4530	1240	1320	1140
10 RMS	4100	5040	2660	2310	3600	3610	1160	1280	1080
20 RMS	5580	6930	4750	2800	4370	1000	990	1200	1120
40 RMS	4070	3920	4240	2440	2940	3040	1000	1000	822
80 RMS	5300	4210	5100	2320	4710	1250	1130	1060	830
110 RMS	4450	4080	3630	3280	2000	590	940	970	760
150 RMS	5750	5510	5320	1610	2470	1800	860	1120	770
FGLP	7590	5210	7300	4440	4160	1400	975	940	975
FGHP	6930	7160	5560	1540	5150	4260	1050	1040	1060
CGLP	5110	6720	4710	5250	4770	4730	925	1230	1180
CGHP	6110	5760	6770	5480	5270	5070	1230	965	1130

B. Exo-Electron Emission

Subsequent investigations by Dr. George Martin have shown the need for incident electromagnetic energy to stimulate exo-electron emission during fatigue cycling. (Reference 1). In these experiments his staff used the Channeltron electron multiplier by itself, without the cylindrical electrostatic mirror. Results with this technique were satisfactory for his purposes.

These successes, coupled with the deduced advantages this approach holds for substrate surface characterization, serve to encourage further development in the face of restrictions imposed by high vacuum.

The equipment necessary for creating the desired high vacuums, while allowing sufficient working room to perform point-to-point and scan measurements, must be designed and costed. Remaining effort on this approach within the current funding period, will be to analyze such designs and costs and the cost of the electron-detection system.

C. NDT Surface Characterization

1. Defining a Surface

Having observed that the various literature definitions for a "surface" are either too idealized, observationally vague, or mathematically restricted, we have sought to define a surface which has the capability to perform the material-energy interactions already cataloged in the sciences of physics and chemistry. This view prepares the way for selection of energy forms useful to the nondestructive characterization of surfaces. Our definition is presented in Figure 2. It is compatible with current thinking in quantum mechanics, particularly with reference to chemical bonding (Reference 2).

A surface is defined here as, "A region between two different phases which exists in an excited, high-energy state relative to the energy states normally associated with each of the bulk phases, where dimensional aspects are quasi-stable but energy aspects are highly mobile."

With this definition we have expanded and clarified, to some degree, the definition of "surface" as stated in Quarterly Report No. 3. Most important in this new definition is that no attempt has been made, as yet, to identify the frequencies and locations (i.e. atomic orbitals, molecular orbitals) of specific energy quantities, such as those which control the wetting phenomenon. Although pinpointing such energy states is beyond present quantum mechanical computational capabilities, awareness of the existence of such states is extremely valuable to this program.

2. Gas-phase Ultrasonic Transmission

Following the concept of deBroglies' "pilot waves" associated with total-internal light reflection, the analogous "pilot waves" in ultrasonic acoustics serve as the basis for the "gas-phase ultrasonic transmission method." It is known in acoustics that a solid-gas interface (surface) presents a huge acoustic impedance (ρV) mismatch, resulting in total internal reflection of the acoustic wave (or pulse)in the solid. This serves as the basis for pulse-echo measurements in nondestructive testing. The "total" reflection is for all practical purposes total energy return, yet a small but significant amount of the acoustic energy is actually transmitted through the surface into the

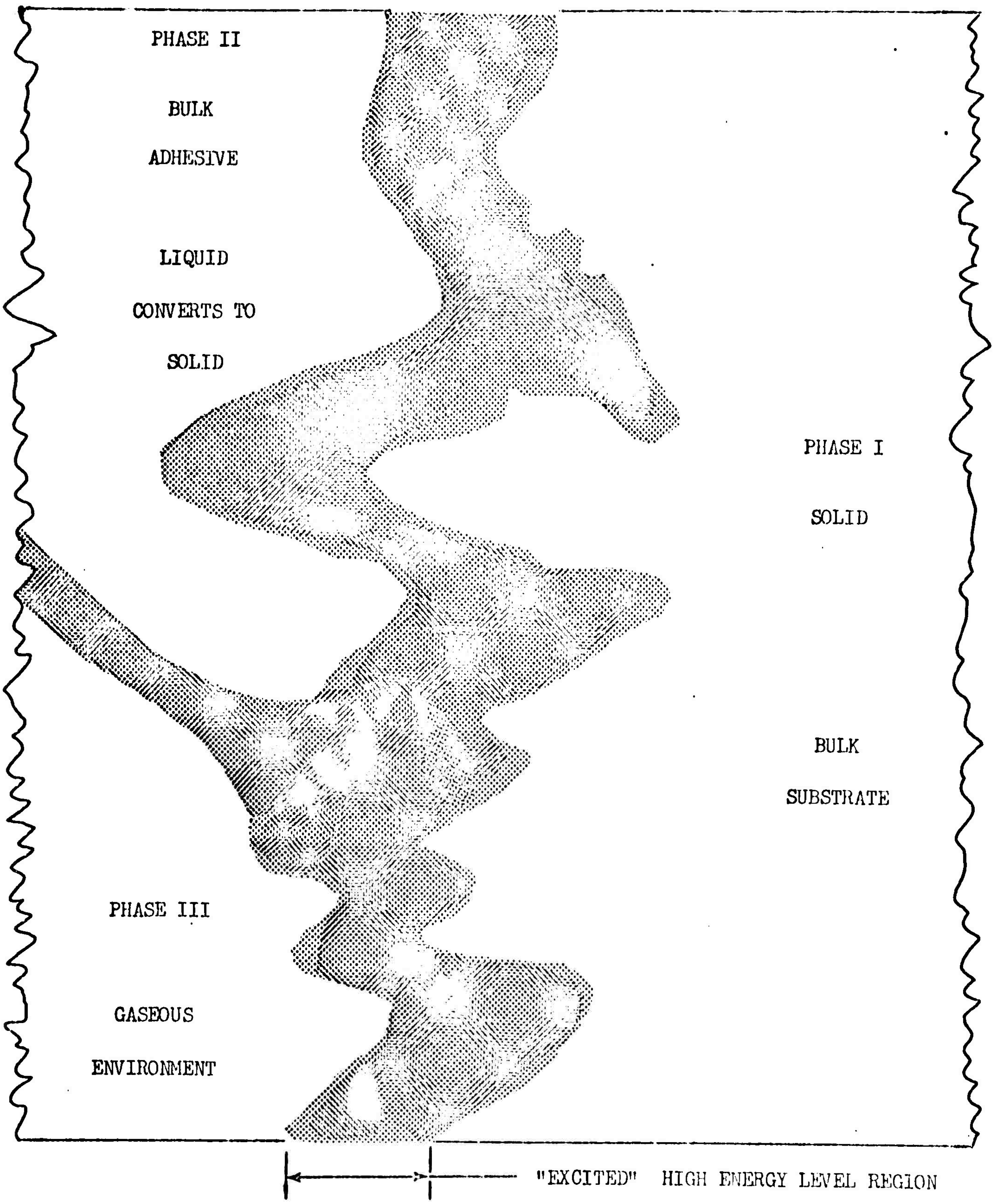


FIGURE 2. CONCEPT OF "SURFACE" IN TERMS OF QUANTUM MECHANICAL ENERGY STATES

gas phase as pilot waves or wave fronts. That amount, and its frequency/phase/amplitude character, must depend upon the energy state (excitement) of the surface. Thus, by measuring the gas-phase transmitted acoustic energy, information concerning the state of the surface will be obtained. Means to experimentally accomplish such measurements are currently being developed.

3. Electric Field Reflectometry

Just as we characterize surface-reflected light in terms of its intensity, color (frequency), polarization plane angle, etc. to describe the character of a surface (i.e. mirror, paint-job, velvet, frost), the reflections of electromagnetic energy at other frequencies will carry information related to surface energetics. By using single-frequency (coherent) energy forms proper characterizations of incident and reflected energy may be readily accomplished. Selection of the frequency provides the means for isolating individual surface material-energy interaction relationships specifically related to adhesive bonding.

By way of a starting point with existing equipment, electric field reflectometric measurements are being conducted at 9.8 GHz (microwaves) and 1 KHz (low frequency capacitance) Figures 3 and 4. The wavelengths associated with these frequencies are orders of magnitude greater than light wavelengths, such that a rough surface does not create the "optical illusion" diffraction effects usually perceived by the casual observer in looking at such surfaces. The specific means of separating first order interactions from the second and third-order interactions of interest are now being investigated.

III. CONCLUSIONS AND RECOMMENDATIONS

None

IV. FUTURE WORK PLANNED

The following work is planned for the quarter 1970 January 20 through 1970 April 19:

1. Complete correlation and analysis of data from surface character study.

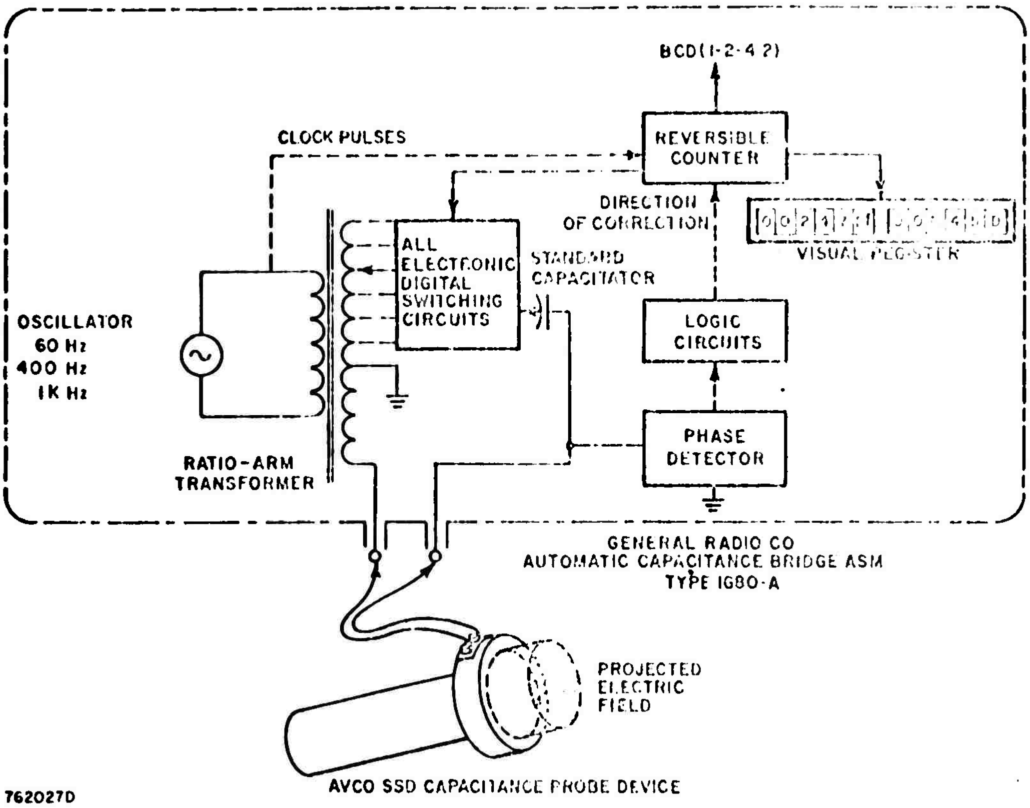
2. Explore feasibility of gas-phase ultrasonic transmission method through initial experiments.

3. Develop means for performing electric field reflectometry at 9.8 GHz and 1 KHz.

John R. Zurbrick-Principal Investigator

E. A. Proudfoot-Program Manager

A



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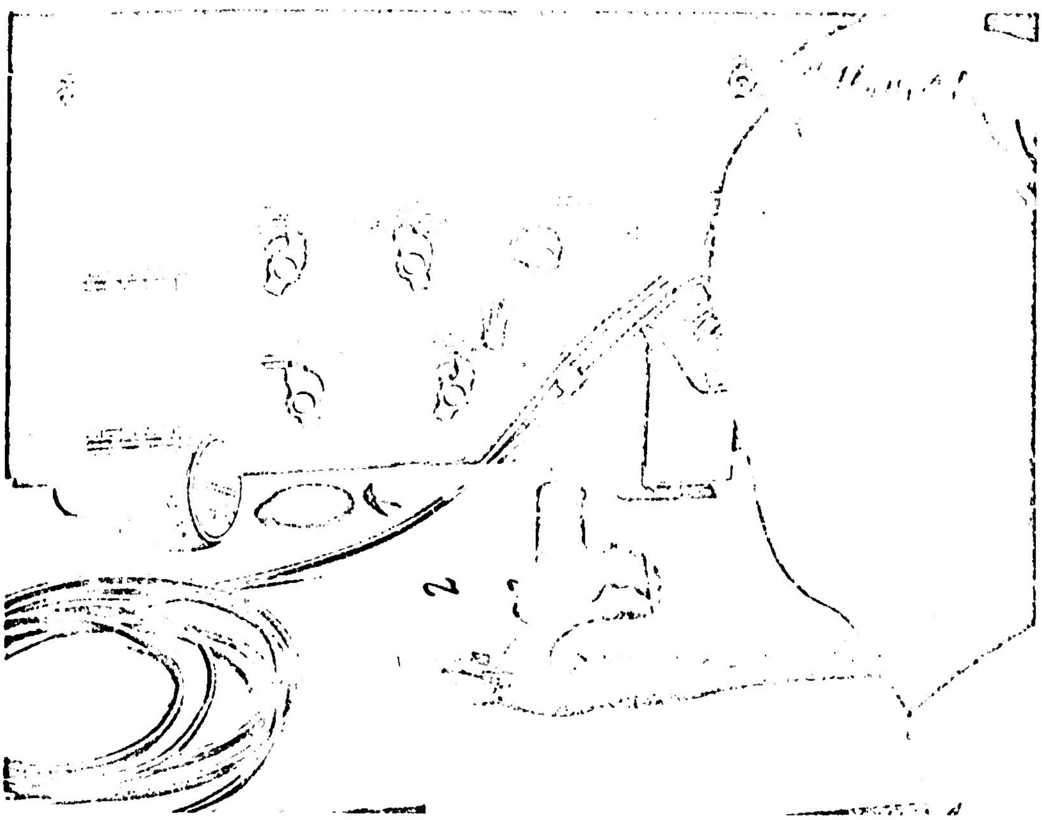

FIGURE 3. LOW-FREQUENCY CAPACITANCE REFLECTOMETRY, 1 KHz

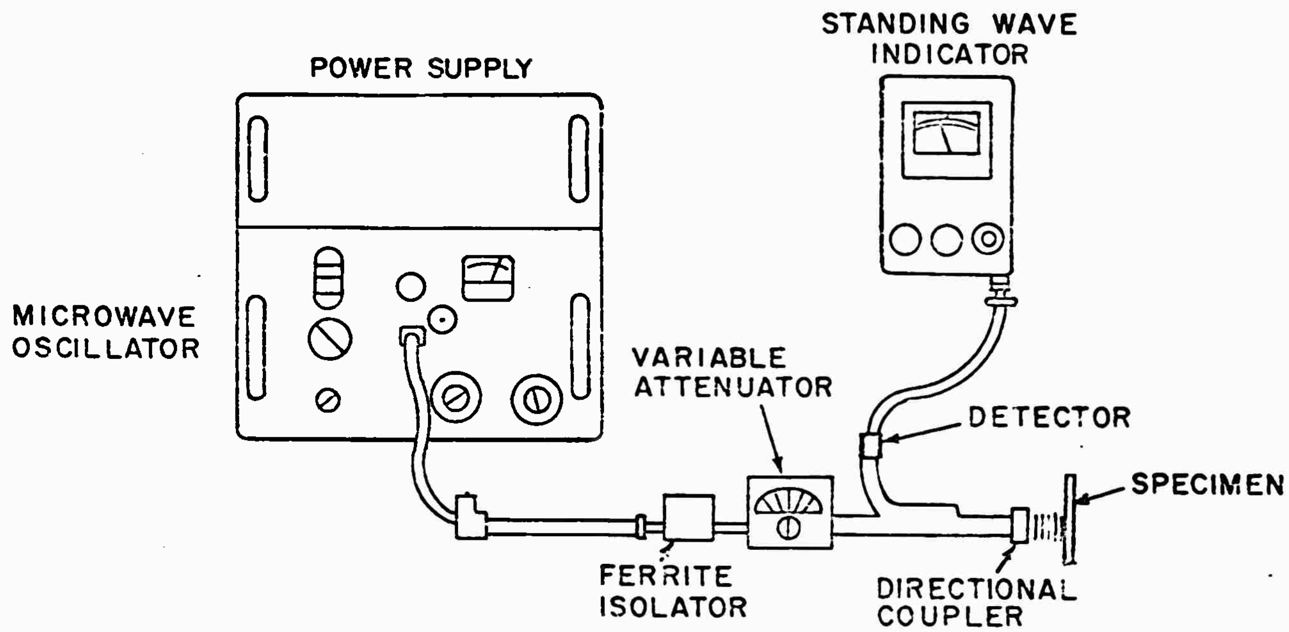

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FIGURE 4. MICROWAVE REFLECTOMETRY, 9.8 GHz.

REFERENCES

1. Martin, G., et al, "The Early Detection of Fatigue Damage" Second Semiannual Report, ARPA Order No. 1244, Report No. NA-69-701, North American Rockwell Corp., 1969 August 31.

2. Pimentel, G. C., and Spratley, R. D., Chemical Bonding Clarified Through Quantum Mechanics, Holden-Day, Inc., Inc., San Francisco, 1969.